THE JOURNAL

OF THE

AMERICAN LEATHER CHEMISTS ASSOCIATION

VOL. XLIX NOVEMBER 1954 No. 11

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SUBSCRIPTION: Members $12.00 a Year Non-Members $15.00

Published Monthly by

THE AMERICAN LEATHER CHEMISTS ASSOCIATION

PUBLICATION OFFICE, EASTON, PA.

Entered as Second-Class Matter at Post Office, Easton, Pa.
Acceptance for Mailing at Special Rate of Postage as Provided for in Section 1103.
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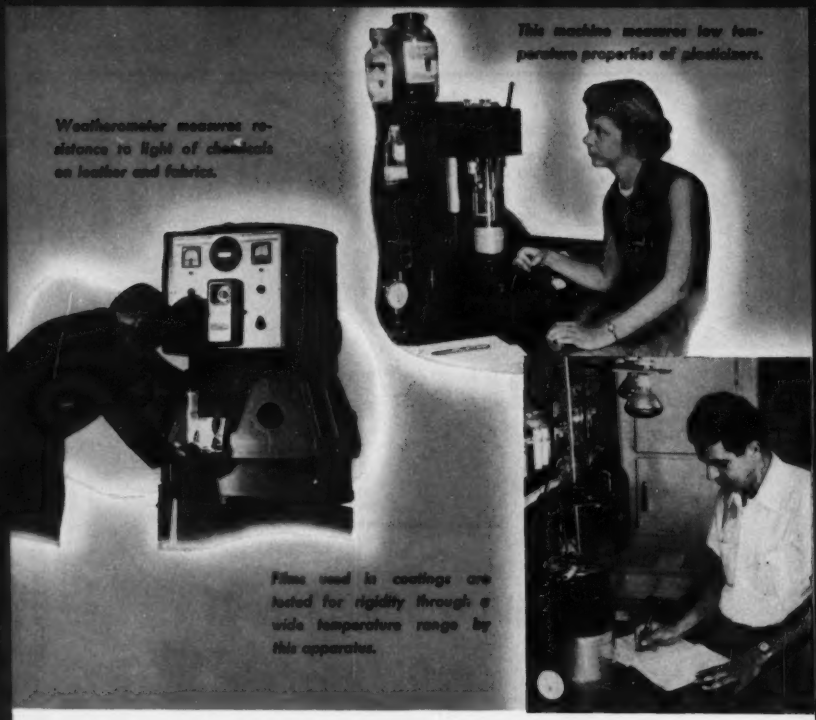
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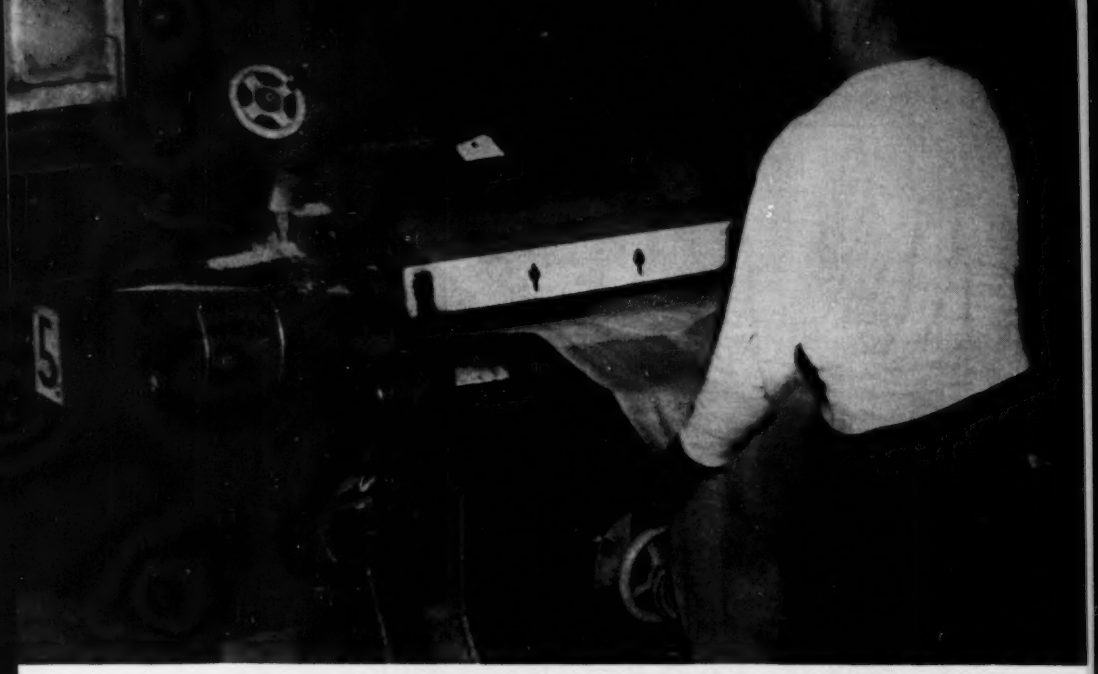

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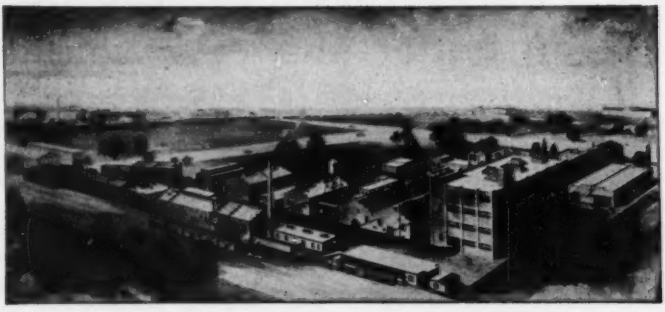

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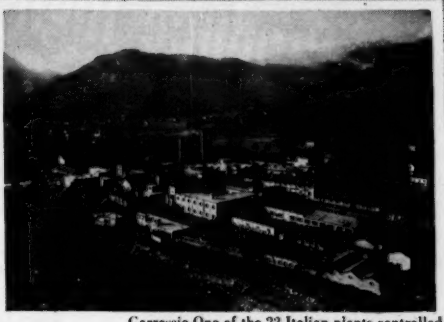

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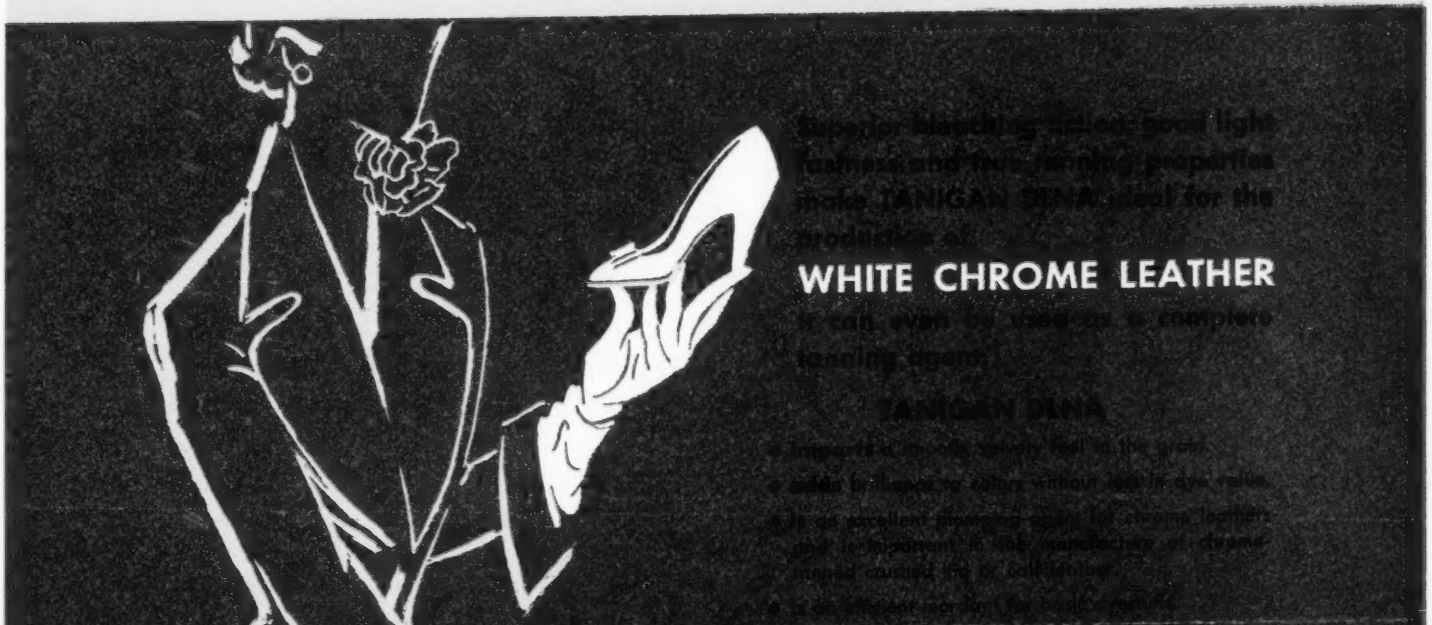

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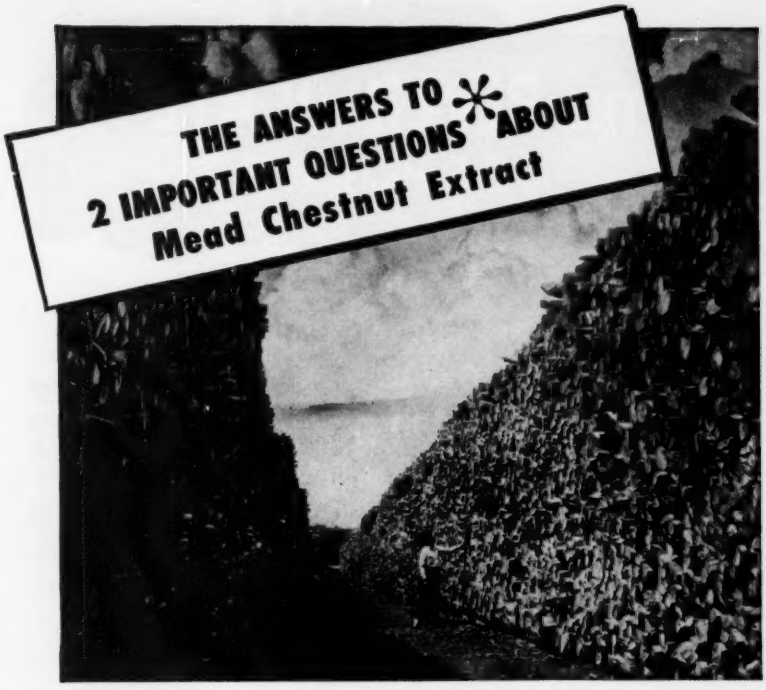

This is only a portion of the many stockpiles of chestnut logs that insure a continuous supply of Chestnut Extract.

1. Is the supply of Mead Chestnut Extract adequate?

Decidedly yes. Recent surveys show that there is enough chestnut wood in our forests to serve the leather industry for many years to come. Huge chestnut stockpiles, such as the one shown above, are converted under chemical control at five extract plants, insuring a steady supply of Mead Chestnut Extract.

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C. Mead Chestnut Extract eliminates up to 28% of leachhouse losses.

D. There are no hidden costs. When you use Mead Chestnut Extract, no time- and money-consuming preparation is required.

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IT'S A FACT!

Mead Chestnut Extract will produce more pounds of plump leather per unit of tannin than any other tanning material.

Vegetable Tannin	Tan Yard Yield (*white weight basis*)
Chestnut Wood Extract	67%
Tanning Material A	61%
Tanning Material B	63%

Calculate your tanning material costs on a leather basis as well as on a tan unit basis. Mead Chestnut Extract, producing four to six pounds more leather, means positive savings.

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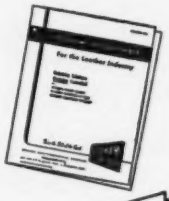

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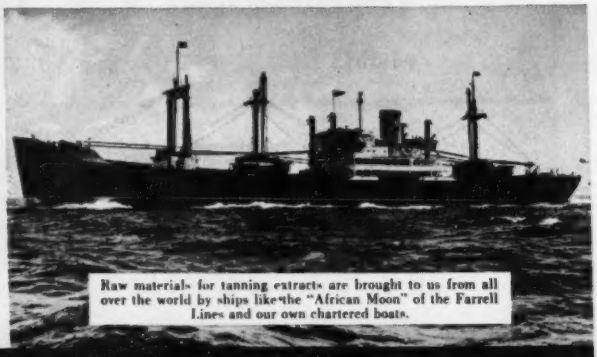

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VOL. XLIX NOVEMBER 1954 No. 11

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Subscriptions—Members, $12.00 annually; Non-members, $15.00 annually.

Published monthly by the American Leather Chemists Association.

Entered as Second-class Matter at the Post Office at Easton, Pa.

Acceptance for mailing at special rate of postage provided for in section 1103, Act of October 3, 1917, authorized July 16, 1918.
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J. R. Kanagy, 9922 Sutherland Road, Silver Spring 5, Maryland.

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Dominic Meo, Jr., Salem Oil and Grease Company, Salem, Massachusetts.

CORRECTION

The following correction is to be made in the September 1954 Journal, page 638, reference number 9. The page number of the reference should be 624 instead of 585.

SECRETARY'S NOTICE

"Manufacturer interested in acquiring formulae for manufacture of low cost resin and plastic finishes for leather on outright purchase or other basis. Address Box AP1900, c/o Secretary, American Leather Chemists Association, University of Cincinnati, Cincinnati 21, Ohio."

Status of the Association as of February 1, 1954

MEMBERS:			
Active	467	New in 1953	73
Associate	353	Lost in 1953	41
Student	4		
	824	Gain	32
SUBSCRIBERS:			
Domestic	82	New in 1953	54
Foreign	169	Lost in 1953	89
	251	Loss	35
MUTUAL MEMBERS:			
(S.L.T.C.)	155	Loss in 1953	2
EXCHANGE SUBSCRIPTIONS:			
Domestic	13		
Foreign	42	Same as 1952	
Canadian	1		
	56		
COPIES TO ADVERTISERS:			
	92	Decrease in 1953	3
TOTAL JOURNAL Circulation	1,378	Decrease in 1953	8

Circulation Breakdown

	United States	Foreign	Canadian	Total
Active Members	399	41	27	467
Associate Members	300	38	15	353
Student Members	2	2	—	4
Mutual Members	—	155	—	155
Subscribers	78	169	4	251
Exchange	13	42	1	56
Advertisers	92	—	—	92
Total	884	447	47	1,378

Members Breakdown

	Active	Associate	Student	Total
United States	399	300	2	701
Canada	27	15	—	42
Foreign	41	38	2	81
Total	467	353	4	824

	Members Breakdown			
	Active	Associate	Student	Total
Maine	—	—	—	—
New Hampshire	8	4	—	12
Vermont	1	—	—	1
Massachusetts	57	59	1	117
Rhode Island	1	—	—	1
Connecticut	2	5	—	7
New York	48	43	—	91
New Jersey	56	43	—	99
Pennsylvania	59	41	—	100
Delaware	13	7	—	20
Maryland	8	6	1	15
Washington, D. C.	10	—	—	10
Virginia	10	1	—	11
W. Virginia	1	3	—	4
North Carolina	3	2	—	5
South Carolina	—	—	—	—
Georgia	3	2	—	5
Florida	—	—	—	—
Kentucky	—	—	—	—
Tennessee	8	2	—	10
Alabama	—	—	—	—
Mississippi	—	—	—	—
Ohio	23	12	—	35
Indiana	2	4	—	6
Illinois	24	21	—	45
Michigan	13	6	—	19
Wisconsin	33	26	—	59
Minnesota	4	—	—	4
Iowa	—	2	—	2
Missouri	7	6	—	13
Arkansas	—	—	—	—
Louisiana	—	1	—	1
North Dakota	—	—	—	—
South Dakota	—	—	—	—
Nebraska	—	—	—	—
Kansas	1	—	—	1
Oklahoma	—	—	—	—
Texas	1	—	—	1
Montana	—	—	—	—
Idaho	—	—	—	—
Wyoming	—	—	—	—
Colorado	—	—	—	—
New Mexico	—	—	—	—
Arizona	—	—	—	—
Utah	—	—	—	—
Nevada	—	—	—	—
Washington	1	—	—	1
Oregon	—	—	—	—
California	2	4	—	6
Total	399	300	2	701

Foreign Members Breakdown

	Active	Associate	Student	Total
Denmark	1	—	—	1
Israel	1	—	—	1
India	3	2	—	5
Cuba	2	4	—	6
Belgium	—	1	—	1
Brazil	5	1	—	6
South Africa	1	2	—	3
Switzerland	2	1	—	3
France	1	3	—	4
Norway	1	1	—	2
Italy	—	1	—	1
Argentina	4	2	—	6
Mexico	2	2	—	4
Greece	—	1	—	1
England	2	1	—	3
Germany	—	1	—	1
Rhodesia	1	—	—	1
Philippines	2	1	—	3
Australia	—	1	—	1
Peru	1	—	1	2
Sweden	2	2	—	4
Uruguay	—	1	—	1
Chile	—	1	—	1
New Zealand	1	1	—	2
Equador	1	—	—	1
Turkey	2	2	—	4
Japan	4	1	—	5
Colombia	—	3	1	4
Puerto Rico	—	1	—	1
Ethiopia	—	1	—	1
Luxenburg	1	—	—	1
Panama	1	—	—	1
	—	—	—	—
Total	41	38	2	81
Canada	27	15	—	42

Exchange List Breakdown

United States	Foreign	Canadian	Total
13	42	1	56

Advertisers — Free Copies

United States	92

S.L.T.C. Mutual Members Breakdown

England	98	Erie	2
Australia	29	Pakistan	1
Sweden	3	Egypt	1
Ireland	4	New Zealand	3
Scotland	3	India	1
Sudan	1	Spain	1
Belgium	2	South Africa	5
Greece	1		
		Total	155

Foreign Subscribers Breakdown

Israel	2	Uruguay	2
Australia	18	Chile	1
Italy	11	Colombia	1
Argentina	5	Brazil	3
India	4	Pakistan	1
Portugal	2	Malaya	1
Germany	16	Siam	1
Russia	6	England	7
Switzerland	3	New Zealand	2
Belgium	2	Spain	1
Sweden	12	Scotland	2
Holland	3	Japan	23
Norway	4	Indonesia	1
Hungary	9	Finland	4
Mexico	1	Peru	1
South Africa	4	Philippines	1
France	11	Austria	2
Denmark	2		
		Total	169

United States Subscribers Breakdown

New York	18	North Carolina	1
Delaware	2	Oregon	1
Massachusetts	7	Missouri	2
Illinois	4	Washington, D. C.	4
Minnesota	3	Wisconsin	1
Pennsylvania	11	Washington	2
Maryland	1	Alabama	1
New Jersey	4	Kentucky	1
California	6	Michigan	2
Ohio	3	New Hampshire	1
Georgia	1	Iowa	1
Indiana	1		
		Total	78

Canadian Subscribers 4

Determination of Amide Nitrogen in Collagen and Other Proteins*

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ABSTRACT

An alkaline hydrolysis method for the determination of amide nitrogen is described. Values obtained by this method compare favorably with the literature values for a number of proteins. A study of the amide nitrogen content of collagen obtained from the hides of identical twin cattle subjected to different nutritional levels shows that the amide value of the collagen is not affected in these experiments. Some changes in amide values of collagen and gelatin due to processing are indicated.

INTRODUCTION

Amide nitrogen is generally determined by the acid hydrolysis methods of either Shore et al.[1] or Gordon et al.[2]. These methods trap the liberated ammonia in the hydrolyzing acid from which it must be liberated by strong alkali and distilled into a medium in which it may be estimated by titration. The kinetics of this method have recently been studied by Cassel and MacKenna[3]. Warner and Cannan[4] proposed an alkaline hydrolysis method which was carried out in a Conway vessel[5]. The liberated ammonia diffused from the hydrolysis medium to the standard acid titrating medium which was enclosed within the same vessel.

These three methods require a considerable amount of work to demonstrate that all of the amide nitrogen has been liberated from the sample because a separate sample must be run for each point on the time curve. The present paper reports an adaptation of the Warner method wherein the procedure is simplified by using a train of Sobel microaeration tubes[6] instead of the Conway vessels.

*Presented at the fiftieth annual meeting of the American Leather Chemists Association, Bedford, Pa., June 7, 1954.

Details of Method

The determination is carried out in a train of Sobel microaeration tubes as shown in Figure 1. The first tube which has the bubbling side arm open to

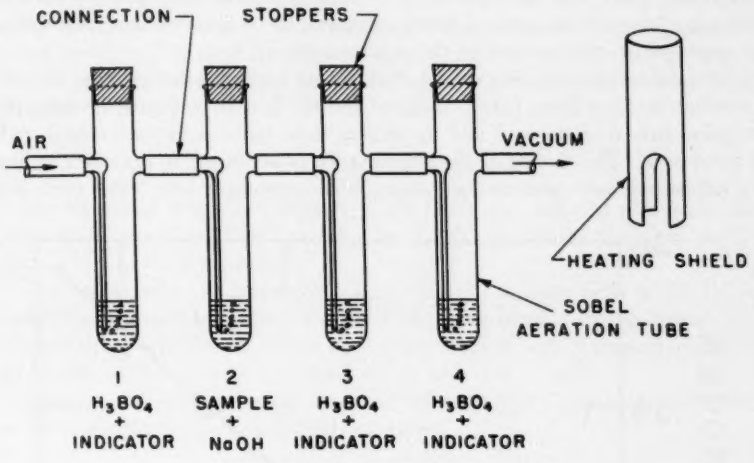

FIGURE 1. Amide Nitrogen Apparatus.

the atmosphere contains 1.5 ml. of boric acid-indicator solution. This tube prevents atmospheric ammonia from influencing the results. The second tube contains the sample, which should contain about 0.3 mg. of amide nitrogen, 3 drops of silicone antifoam solution, and 2 ml. of 2.0 *N* sodium hydroxide solution which was added at the start of the determination. The third and fourth tubes each contain 1.5 ml. of boric acid-indicator solution.

The tubes are connected with 1.25 inch long sections of $^3/_{16}$ inch bore rubber tubing so that the bubbling side arm of each tube is connected to the previous tube. These connections are made and tubes 1, 3 and 4 are stoppered with rubber stoppers before the alkali is added to tube 2. The fourth tube is connected to a vacuum source through a flow regulating device so that an air flow of about 120 bubbles per minute can be maintained throughout the determination. A very fine capillary several inches in length has been found most suitable for producing the desired regulation. The ends of the capillary must be protected from dust by glass wool or other plugs. The vacuum should be connected just prior to introducing the alkali to tube 2 and tube 2 should be stoppered immediately after the alkali is introduced.

The tubes are then set into a rack which straddles the side of a 70° water bath so that the first two tubes (the pretrap and reactor) are immersed in the bath and the last two tubes (the absorbers) are hanging in air at room temperature. The reactor tube is then covered with a section of slotted copper tubing which extends from the top of the reactor tube down to one-half inch below the water level of the bath. This tube conducts heat from the bath to the top of the reactor and prevents condensation of moisture and adsorption of ammonia in this portion of the reactor tube.

At the desired time intervals (2, 3, 4, 5 and 6 hours after starting the determination have been found suitable) the air flow is stopped, the first absorption tube disconnected and the tubing from the reactor tube closed with a pinchcock. The closing of the reactor tube is necessary to prevent the loss by diffusion of any ammonia remaining in the connector tube. The ammonia

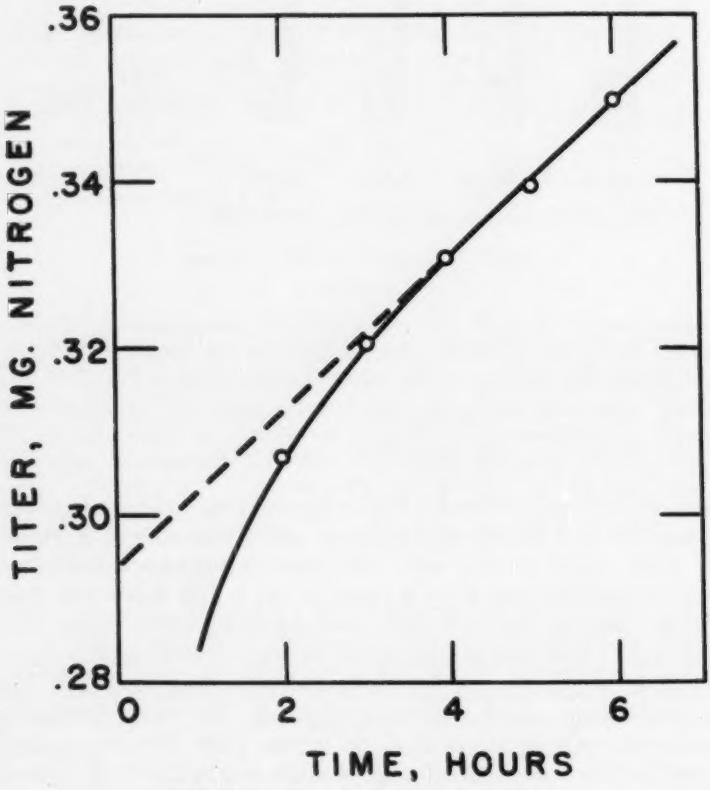

FIGURE 2. The rate of Ammonia liberation.

trapped in the absorption tube is then titrated with 0.1 *N* hydrochloric acid. A one ml. capacity microburette is used for this titration. A blank tube containing only the boric acid-indicator solution may be used to match the indicator end point color where the highest accuracy is desired. The indicator color in the second absorption tube usually will not change. However, should there be a change in color this tube must be titrated also. The absorption tubes are returned to the train and the air flow resumed. The accumulated titers are plotted against the times (Figure 2) and the straight line portion of the curve is extrapolated to zero time to give the titer due to the amide nitrogen liberated.

Special Reagents

Sobel's mixed indicator—8 parts of a 0.1 per cent solution of bromocresol green in 95 per cent alcohol and 1 part of a 0.1 per cent solution of methyl red in 95 per cent alcohol.

Boric acid-indicator solution—2.0 gm. of boric acid crystals and a 1 ml. of Sobel's mixed indicator are made to 100 ml. with water. The indicator color is adjusted to a very faint purple with a few drops of 0.01 *N* sodium hydroxide when the volume is within about 2 ml. of the final volume.

Silicone anti-foam solution—is made by suspending about one-half gram of silicone anti-foam-A in 100 ml. of ether.

Experimental Verification

The method was employed to determine the amide nitrogen of a number of proteins including casein, edestin, β-lactoglobulin, ovalbumin and serumalbumin. All of these were of the purest grade available commercially for laboratory work. Table I shows that the results obtained with this proposed

TABLE I

The Amide Nitrogen Content of Various Proteins

Protein	Determined % of total N	Literature % of total Nitrogen	Reference
Casein	10.7 ± 0.14 (8)*	10.2	7
		9.1	8
Edestin	10.7 ± 0.15 (17)	10.3	4
		9.5	8
β–lactoglobulin	6.8 ± 0.21 (12)	6.9	4
		6.9	8
		6.9	9
Ovalbumin	7.0 ± 0.22 (11)	6.2	1
		6.6	8
Bovine Serum Albumin	5.8 ± 0.17 (6)	5.6	9

*The standard deviation calculated for the number of determinations given in the brackets.

method agree very well with previous values reported in the literature. A standard error of around 0.2 was obtained for each protein where sufficient experimental results had been collected to enable the calculation of the standard error. The method, therefore, seems to be suitable for determining or following changes in amide content of proteins in general.

Collagen and Gelatin Studies

The method was then applied to determine the amide nitrogen content of a number of collagen and gelatin preparations. The results are shown in Table II. In general the amide values center around 3.6 per cent of the total nitrogen. The three major exceptions are the American standard hide powder

TABLE II

The Amide Nitrogen Content of Various Collagen and Gelatin Preparations

Sample	Description	% Total Nitrogen	% of N as Amide
1	American std. hide powder	17.87	2.41
2	Limed "white" hide	17.63	2.61
3	Purified collagen (Cassel)	18.05	3.93
4	Defatted salted hide	16.90	3.79
22	Twin hides—normal diet	17.83	3.72
21	—restricted diet	18.05	3.80
19	Twin hides—normal diet	18.20	3.28
20	—restricted diet	18.03	3.41
31	Twin hides—normal diet	18.28	3.64
32	—restricted diet	18.13	3.66
8	Purified collagen	17.93	3.72
8a	8-Ether alcohol extd.	18.22	3.54
8b	Gelatin from 8a	18.61	3.17
9	Eastman pig skin gelatin	17.82	3.55
10	Eastman calf skin gelatin	17.15	0.42

which gives a value of 2.4 per cent, limed hide (in the white from a commercial tannery), which gives a value of 2.6 per cent, and the Eastman calf skin gelatin with an isoelectric point of 4.7, which gives a value of 0.42 per cent.

Sample 3 which was prepared by Dr. J. M. Cassel as a purified collagen has the highest amide value. The comparatively mild treatment received by this sample during its preparation would indicate that the amide value of 3.9 per cent of the total nitrogen should be close to the maximum value to be expected for collagen. Cassel and Kanagy [10] have proposed the value of 3.8 per cent in their specification for purified collagen. The amide value for a sample (No. 4) taken from a defatted salted hide also had a value of 3.8 per cent.

The values for the standard hide powder and the limed "white" hide are in close agreement and the difference between these values and those of the purified collagen figures is probably due mainly to the liming treatment to which each of these materials had been subjected during their preparation.

Samples 19, 20, 21, 22, 31 and 32 are from the center split of the bend portions of hides taken from three pair of identical (monozygotic) twin cattle which had been subjected to nutritional studies[11, 12]. One twin was kept on a standard diet and sacrificed at a given weight. The other twin was put on a substantially reduced diet (deficient only in calories) for about 90 days and then fed to regain the lost weight and was sacrificed at an equal degree of fatness as the control twin. The sample numbers of the hides in Table II correspond to the animal numbers in reference 12 where details of the feeding experiments are described. These three pairs of hides were salted and preserved until the time they were worked up into the purified collagen. The six hides were extracted simultaneously in the same tumbler so that all treatments could be called comparable. They were washed with water and 8 per cent salt solution, fleshed, clipped and split to obtain the center layer. The center layer was then extracted 3 times each with 8 per cent salt, acetone, water, half saturated lime, water, dilute acetic acid, water and acetone. The samples were then air dried and ground in a Wiley mill. The close agreement of both the total nitrogen and the amide values for the collagen from the hide of each pair of twin animals would indicate that the reduced diet did not result in the production of an altered collagen. This is in agreement with the study performed by Lightfoot and Coolidge[13] on guinea pigs. They concluded that collagen continues to be produced in growing animals at the same rate in fasting animals as in well fed animals and that collagen formation is quantitatively the most important bioligical reaction using nitrogen for growth.

The slight variations in the amide values between the pairs of twins might be due to a number of factors but the data are not adequate to permit a proper choice among them.

The magnitude of the changes in amide values produced by fairly mild treatments is shown in the samples numbered 8. The original collagen (No. 8) was prepared from a fresh hide by extraction with 10 per cent salt solution, acetone, half saturated lime water, and dilute acetic acid. It gives nitrogen and amide values close to those of the best purified collagens. Extraction of this collagen with ether and warm alcohol followed by water (8a) has reduced the amide value by 0.2 units. Extraction of this ether extracted collagen with water at pH 7.0 and 120°C. for 30 minutes produced a gelatin (8b) which had an amide value 0.3 unit lower or 0.5 units lower than the original collagen.

The two commercial purified gelatins (samples 9 and 10) show the wide variation of amide values which it is possible to obtain without too much degradation to the gelatin molecule.

These examples show that the alkaline hydrolysis method for determining amide content of proteins can be carried out in a simple fashion to obtain values comparable with those in the literature. When the method was applied to a study of the purified collagens obtained from a series of identical twin cattle subjected to nutritional experiments the data showed that the amide content of the collagen did not depend upon the nutritional history of the animal if the animal was in a satisfactory nutritional state when sacrificed.

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Received April 1, 1954.

Discussion

Robert M. Lollar: I think this is a very interesting paper because the literature is all too deficient on information in the variations in any of the properties of collagen which occur in different animals from the same species. I find the comparative results on the three sets of identical twins quite interesting and only wish we had more of such information.

There were certain factors in connection with the work that I thought we might elaborate on a little further. One: in commenting on the differences between the hide powder and the collagen secured from the hides, you noted the difference in the rate of solution. And in the abstract, the term "hydrolysis" is used.

I should like to ask Dr. Hoover if he would care to comment on how far he thinks the breakdown into amino acids has gone during the period of time that the amide nitrogen is being released.

S. R. HOOVER: I believe about half of the free amino nitrogen is released in the six hours. There is not a great deal of free amino nitrogen released in the first hour when the amide nitrogen is complete. It is on the order of five to ten per cent by the time the first hour is up. But then that goes ahead. This is at 70 degrees. At 100 degrees you can get almost complete hydrolysis in six hours.

LOLLAR: That, then, takes me to a part of the next question, which is concerned with the comparative accuracy of the values secured on collagen and the values secured on the other proteins.

First, I note that the level of amide nitrogen is appreciably lower in the case of collagen than it is on the other proteins. We often find that the standard deviation bears some relationship to the magnitude of the value being measured, and I think of that particularly in connection with some work we have done in connection with the free amino nitrogen technique on incompletely hydrolysized proteins where we find the error term of this extrapolated value is much larger where the degree of hydrolysis is small.

I did not see in the slides or the paper, as I had a chance to look over it, any standard deviation which specifically applied to collagen.

HOOVER: I was waiting to see if it was there, too.

The answer is that the standard deviation is essentially the same for the different materials and the reason that is true is that we adjust our sample size so that we get the same amount of ammonia off from the various samples. In the collagen we took three times as much material to run as we did with casein, but that does not cause any complications. So that I do not believe there is reason to expect any significant difference in error in a sample that is low in amide nitrogen. Actually, Mr. Viola usually runs a pilot to decide how much sample he is going to take if he cannot guess fairly well what his answer is going to be.

We try to shoot at about this level of 350 to 500 microliters, half a milliliter, of titration.

LOLLAR: In other words, the standard deviation of an individual value, whether it be on collagen or on one of the other proteins, is essentially the same?

HOOVER: Yes.

LOLLAR: If we then take this information and apply it to some of the differences as, for instance, the difference between the more native collagen and limed collagen (if I can remember the tables correctly, the difference is just roughly six standard deviations) it indicates to me that rather large differences would have to be secured for any small size experiment. How many replicate analyses are represented, for instance, in a comparison between the native collagens and the limed collagen?

HOOVER: Each point is a mean of three results. And in general those three results actually agree within about a tenth of a per cent. Now, I do not think we have pushed this, as I said in my original apologia, we have not pushed this to the limit that can be done for precise comparisons of the type you have in mind. I think the standard deviation can be held to underneath a tenth of a per cent.

LOLLAR: I was interested, for instance, in the three sets of twins, that all three sets of the restricted twins had actually a higher ratio of amide nitrogen. But that is not significant with the error encountered in the experiment at hand except that three coincidences has a certain significance.

LUDWIG SELIGSBERGER: (QM R. & D Command): In a paper in 1937 Kuentzel* mentions that he boiled collagen in NaOH, and determined the insoluble matter which he called elastin at that time. He found, of course, much more in the flesh layer than in the corium. Have you observed anything insoluble in this experiment which you could possibly determine? Would you expect that layerwise analysis would lead to differences in the amino nitrogen? Also, what is your opinion as to a possibility to differentiate between collagen and elastin?

HOOVER: I believe you made three points.

The first is that in this work reported here, we had the middle split. The corium was what we were working on in all cases except the white hide which was a regular white hide. We have done a great deal of work in connection with the purification of collagen. We have not been at it very long but have done a great deal of work in that time, trying to get the differential resistance to alkali on some reasonable basis. Frankly, Dr. Seligsberger, we have not. The collagen-elastin argument is a mystery as far as I am concerned.

Your third point?

SELIGSBERGER: Have you visually observed anything insoluble in these experiments?

HOOVER: No. We were using the corium split and it all goes into solution.

DONALD F. HOLLOWAY: One of those was a lime cured gelatin. Was the other acid cured or lime cured?

HOOVER: The answer is yes. One of each. They are Eastman top grades of gelatin and some of you gentlemen may be familiar with them. They worked out and published it in 1929 for the photographic grades and they have not changed one iota the type of processing.

HOLLOWAY: You have one acid cured and one lime cured and that accounts for the difference in the analysis?

*C 1936, 567.

HOOVER: Yes, but it is more than just liming, to get it down there. After all, with hide power and other things we fooled with, you do not get down to the level of 0.4 per cent for the amide nitrogen. I do not know how they get down to such a value.

HOLLOWAY: It was the acid one that had the low value, didn't it?

HOOVER: They claim the acid treated stuff has the high pH. It has the highest iso-electric point.

HOLLOWAY: The low amide nitrogen?

HOOVER: No, it is the one with the high amide nitrogen.

Combination Tannages: Vegetable Tanning of Chrome Tanned Hide Powder and Pelt

By P. HANUMANTA RAO

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INTRODUCTION

Combination tannages are used to produce better leather in the industry. In some cases the rate of tanning is increased, saving operation costs. By combination tannages it is possible to combine the desirable qualities of more than one tannage in the same leather. In addition to this, these tannages throw much interesting light on the nature of the tanning process itself, as to the reactive groups of the tanning agent and collagen, which are involved in the interaction.

In the present paper some studies about the vegetable tanning of chrome tanned leather (hide powder and also pelt) are presented. India has plenty of different natural tanning materials and not all of them are used in tanning practice at present. This is mainly due to two reasons, the first being the lack of systematic study of the nature of these materials and the other, the conservative nature of the leather trade in India with the consequent reluctance to change. Taking this into consideration, a systematic study of some vegetable tanning materials and tanning methods was started at the University of Madras [1]. The present work is a part of the investigations carried out at Stockholm.

Wood [2] reported chromed gelatin fixed as much tannin as gelatin. Wood and Holmes [3] on the other hand found decreased fixation of vegetable tannin by pretreatment of hide with chromium salts. The first systematic investigations in this field were carried out by Gustavson [4] and he found cationic chrome tanning increased the tannin fixation, probably by the activation of the basic groups due to the reaction of acidic carboxyl groups of collagen with the cationic chromium compounds. Anionic chrome tanning decreased the tannin fixation demonstrating the similarity in their interaction with collagen. The acidity of the chromium sulfate in the chrome leather also was reduced showing the readiness with which the constituents of the tannin solutions enter the chromium complex and displace the acido groups. Cationic chrome tanned stock in freshly prepared state was shown to incur loss in the amount of fixed chromium after heavy tannage with vegetable tannins. However, the chrome-collagen compound became stable on ageing. Vegetable tannin fixation by chrome tanned collagen was much less dependent on the pH of the tanning solution, than in the case of untreated collagen. This was explained by taking into consideration the rigidity of the chrome tanned protein, which resisted the swelling effects due to pH variations. These observations have been confirmed by subsequent workers. Page and Holland [5] observed that prechroming collagen increased only the rate of tannin fixation, reducing the tanning period for maximum fixation from a few years to some weeks, but not the absolute amount of tannin fixed. Van Vlimmeren [6] believed that the extent of tannin fixation depended on the water content or swelling of the chromed hide powder. However, in practice it is not common to dry the leather or vary the moisture content before retannage. Even in theoretical studies, it must be remembered that drying brings in complications, which are not relevant to the present problem. Gustavson [7] contends that the chrome tanned hide powder or leather should not be dried before retanning with vegetable tannin as this leads to changes in the reactive groups of collagen.

Experimental

50 g of hide powder (Lyons) was tanned with sulfur dioxide reduced chrome liquor (675 c.c. water and 125 c.c. chrome liquor of 29.0 g/1 Cr_2O_3 and 67 per cent acidity) for 6 hours. The tanned hide powder was filtered and washed. This was kept in a bottle with ground glass stopper, keeping paraformaldehyde in a small test tube as a preservative, to prevent mold growth. The hide powder contained 5.0 per cent Cr_2O_3, 5.74 per cent SO_4, and 88.0 per cent collagen on dry basis, and had a moisture content of 75.5 per cent (24.5 per cent dry matter). The overall acidity of the chromium sulfate in the hide powder was 56.6 per cent, as there was no neutral sulfate present. The per cent acidity of sulfate Cr– complex fixed by collagen as determined

by the pyridine method was 33. Portions of this hide powder and air dry Lyons powder (equivalent to 1.0 g of collagen) were tanned with solutions of tannin (50 c.c. 5 per cent solution of solid extract) from different tanning materials for 48 hours. pH determinations were made on the exhaust solutions. The hide powders were washed with distilled water until the wash-water gave no precipitate with gelatin-salt reagent. They were then dried first for a few hours in the air, later in an electric oven at 110°C overnight and analysed. The results are presented in the following table. Total sulfur was

TABLE I

Material		Exhaust pH	% Coll -agen	% Cr_2O_3 on h.p.	% Cr_2O_3 on Coll.	% SO_4 on h.p.	% SO_4 on Coll.	% Acidity of Cr-coll compound	Degree of tannage (Vegetable).
1. Avaram	Bl	4.4	66.1	——	——	——	——	——	51
"	Cr	2.6	53.3	3.1	5.82	1.44	2.7	24.3	79
2. Konnan	Bl	4.2	63.8	——	——	——	——	——	56
"	Cr	2.7	53.8	3.1	5.77	2.53	4.71	42.6	75
3. Divi	Bl	3.4	51.2	——	——	——	——	——	95
"	Cr	2.3	50.4	3.06	6.07	1.71	3.39	29.5	89
4. Mimosa	Bl	3.4	62.8	——	——	——	——	——	59
	Cr	2.9	52.6	3.30	6.27	0.63	1.20	10.0	83
5. Tannic	Bl	2.7	50.0	——	——	——	——	——	100
acid	Cr	2.2	48.2	2.90	6.02	0.67	1.40	12.1	100
6. Myro-	Bl	5.0	67.4	——	——	——	——	——	48
balan	Cr	3.4	52.8	3.10	5.88	1.27	2.40	21.7	81
7. Valonia	Bl	3.9	65.5	——	——	——	——	——	52
"	Cr	3.4	54.9	3.62	6.60	0.51	0.93	7.3	75
8. Gambier	Bl	4.4	61.0	——	——	——	——	——	64
"	Cr	4.2	57.0	3.37	5.91	0.39	0.69	6.1	69
9. Quebracho	Bl	5.1	60.2	——	——	——	——	——	66
	Cr	3.5	50.2	2.94	5.90	0.99	1.98	17.8	91
10. Sulph.	Bl	5.7	62.1	——	——	——	——	——	61
Quebracho	Cr	4.6	51.2	2.84	5.54	1.03	2.01	19.1	87
11. Oakwood	Bl	4.0	63.9	——	——	——	——	——	56
"	Cr	3.7	53.3	2.69	5.05	0.46	0.87	9.0	82
12. Chestnut	Bl	3.0	55.9	——	——	——	——	——	79
	Cr	2.5	50.8	2.77	5.46	0.78	1.53	14.5	90
13. Canaigre	Bl	5.0	63.4	——	——	——	——	——	57
	Cr	4.6	55.3	2.90	5.25	0.20	0.36	3.6	75
14. Sumac	Bl	4.3	62.1	——	——	——	——	——	61
"	Cr	3.9	52.4	3.20	6.10	0.24	0.45	3.9	84
15. Spruce	Bl	4.3	66.6	——	——	——	——	——	50
"	Cr	4.0	54.1	3.26	6.03	1.07	1.98	17.1	77

determined by the Grote-Krekeler method and correction for sulfur in collagen (0.4 per cent) was applied.

Correction for sulfur is applied when calculating sulfur in Sulfited quebracho tanned hide powder.

The effect of pH on the fixation of tannin by chromed hide powder was studied for a few materials, Avaram, Mimosa, Gambier and Tannic acid. Blank hide powder also was tanned in the same way with the respective tan liquors, at the mentioned pH values. The tan liquors were brought to the said pH values by the addition of *N* HCl or *N* NaOH to a more concentrated solution than was used, and this was diluted and used for the tanning experiments. pH determinations were made on the exhausts. The hide powders were washed as usual until the wash water gave no precipitate with gelatin salt reagent. Analytical data of the hide powders are tabulated below.

TABLE II

Hide Powder				Chrome tanned hide powder									
pH		% Collagen	Degree of tannage	pH		% Collagen	$\%Cr_2O_3$ on h.p.	$\%Cr_2O_3$ on coll.	$\%SO_4$ on h.p.	$\%SO_4$ on coll.	% Acidity of Cr-SO_4 compound	% collagen	Degree of tannage Vegetable
Original	Exhaust			Original	Exhaust								
(a) Avaram													
2.3	2.7	64.8	54.0	2.3	2.5	60.7	2.97	4.86	2.61	4.30	46.6		56
3.4	4.3	64.8	54.0	3.4	3.1	62.6	3.18	5.08	2.38	3.80	39.4		51
6.0	5.5	65.0	53.5	6.0	3.4	61.5	3.06	4.98	2.31	3.75	39.7		54
7.0	6.2	66.8	49.4	7.0	3.9	60.0	3.14	5.23	1.50	2.49	25.1		59
9.0	8.9	78.9	26.5	9.0	8.3	64.5	3.41	5.27	0.00	0.00	0.0		50
(b) Mimosa													
1.6	1.7	58.8	69.7	1.6	1.7	60.0	3.20	5.33	(2.00)	—	—		58
2.6	3.3	61.7	61.8	2.6	2.6	59.7	3.20	5.36	1.74	2.91	28.6		59
4.8	4.9	67.9	47.0	4.8	3.6	58.2	3.20	5.50	1.01	1.74	16.7		65
7.3	6.2	69.0	44.6	7.3	4.3	57.7	3.30	5.72	0.42	0.72	6.6		67
8.1	7.3	68.3	46.1	8.1	6.5	55.6	3.30	5.94	0.00	0.00	0.0		74
9.3	8.9	80.5	23.9	9.3	8.6	62.3	3.60	5.78	0.00	0.00	0.0		55
(c) Gambier													
2.0	2.8	70.3	42.0	2.0	2.6	68.9	2.90	4.20	1.40	2.01	25.2		40
3.0	3.5	70.5	41.6	3.0	3.2	62.6	3.40	5.40	0.69	1.11	10.7		53
4.0	4.4	68.6	45 5	4.0	4.0	63.8	3.10	4.90	0.65	1.02	11.0		51
6.6	5.8	74.3	34.3	6.6	4.9	62.5	3.15	5.04	0.21	0.33	3.5		55
8.0	6.8	76.1	31.1	8.0	6.2	64.6	3.40	5.26	0.00	0.00	0.0		50
(d) Tannic acid													
1.2	1.4	43.6	128.9	1.2	1.4	50.8	2.90	5.71	1.00	1.97	18.0		89
2.7	3.0	59.2	68.6	2.7	2.3	53.3	2.80	5.25	—	—	—		82
3.6	3.7	63.0	58.4	3.6	2.9	53.8	3.20	5.95	1.10	2.04	18.0		78
4.6	4.7	62.7	59.0	4.6	4.0	56.1	3.20	5.70	1.10	1.96	18.0		71
6.0	5.7	58.9	69.4	6.0	4.9	55.0	3.00	5.45	0.20	0.36	3.5		76
7.4	7.2	64.3	55.2	7.4	6.6	58.2	3.30	5.67	0.12	0.21	1.8		66
8.1	7.7	75.8	31.5	8.1	7.5	61.5	3.40	5.53	0.00	0.00	0.0		57
9.1	8.5	82.9	20.2	9.1	8.5	65.5	3.40	5.19	0.00	0.00	0.0		48
10.1	9.0	87.0	14.6	10.1	8.9	68.0	3.60	5.29	0.00	0.00	0.0		42

It is interesting to study the effect of nontannins on the chromed hide powder. Gambier extract, 100 c.c. of 5 per cent solution (obtained by diluting 20 per cent solution 4 times) was detannised with 5.0 g. of American standard hide powder, filtered first through cloth and then through a Buchner funnel and was finally made up to 100 c.c. This solution gave a slight precipitate with gelatin salt reagent. The liquor originally contained 5.3 g. total solubles and 0.17 g. ash per 100 c.c. and the detannised solution contained 2.25 g. total solubles 0.13 g. ash per 100 c.c. The chromed hide powder, equivalent to 1.0 g collagen was treated with 75 c.c. of the detannised solution for 5 days. The hide powder was filtered, washed, dried and analysed. The following table gives the results of analysis.

TABLE III

pH	Detannised liquor 4.4	Exhaust 3.6
	% on h. p.	% on collagen
Cr_2O_3	3.60	4.74
SO_4	0.55	0.72
Collagen	76.0	——
Fixed matter (by difference)	19.85	26.1
% Acidity of Cr—collagen compound	7.9	

Actual tanning experiments were conducted with small pieces of chrome tanned calf skin using avaram, mimosa and gambier. A larger piece also was retanned with avaram, because this is the material that has a special place in the East India tannage. The results are very encouraging, particularly with avaram where the retanned leather has a very nice feel, appearance and color.

Discussion

The results from Table I show that the vegetable tanning of prechromed hide powder does decrease the acidity of the chromium sulfate present, and the extent to which this is done is characteristic of each tanning material, most probably depending on the nature of the nontannins it contains. The increase in tannin fixation by prechroming also seems to depend on the nature of the tannin. All vegetable tannins react with hide protein at least in two ways, ionically with the NH_2 groups and coordinatively at the CO.NH groups. This aspect of the problem has been understood well by studying the reaction of vegetable tannins with hide powder treated with condensed sulfo-acids (certain syntans), that inactivate only the basic groups.[8] This was studied for some Indian tanning materials and presented elsewhere, which gave the proportion of ionically and non-ionically reacting components of tanning material with collagen. However, some tannin, and probably

nontannin too, enters the chromium complex, displacing the sulfate and reducing the acidity of the chromium complex. This is due to the organic acids and their salts present in tan liquors, to a great extent. Konnan and Divi seem to be different in this respect showing little or no displacement. In addition to this the chromium compounds (cationic) mainly react with the carboxyl groups of the collagen and in a way activate the basic amino groups. The chromed hide powder used was aged enough and hence displacement of chromium was not considerable.

Fig. I (a and b) show the effect of pH on tannin fixation graphically.

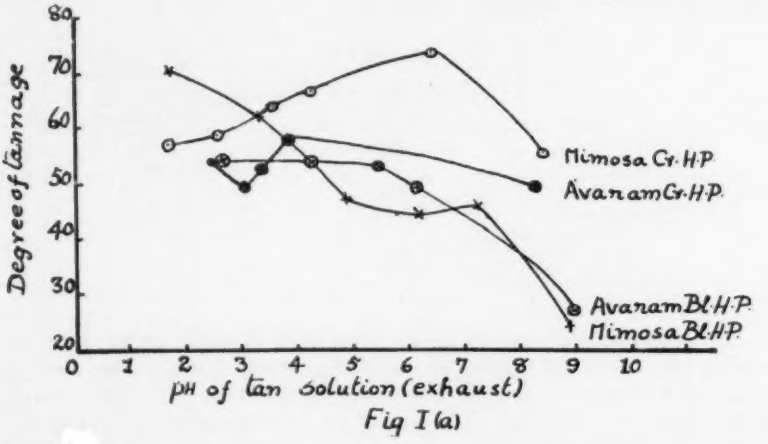

Fig I (a)

The tannin fixation is less pH dependent in the case of chrome tanned hide powder than with the blank hide powder. The degree of tannage at the pH values below 2 or 3, of the chrome tanned hide powder in all the cases is lower than that for blank hide powder. At higher pH values the tannin fixation is always higher for chromed hide powder. However, gambier tannin fixation is not so much pH dependent even with blank hide powder. This might be due to the nature of the tannin fixed, probably mostly by co-ordinative forces predominantly at the peptide groups, which are not affected or activated by changes in pH. The same applies after chrome tanning also, where the increase in degree of tannage is not considerable. Gambier tannin seems to be unable to combine with the amino groups to any great extent. In all other cases the increase in tannin fixation is considerable, except with divi divi where the reason is not known. It might be possible that the organic acids present in this material mostly are less capable of forming complexes with chromium. However this is only a postulate. Further work in this field is planned to be pursued.

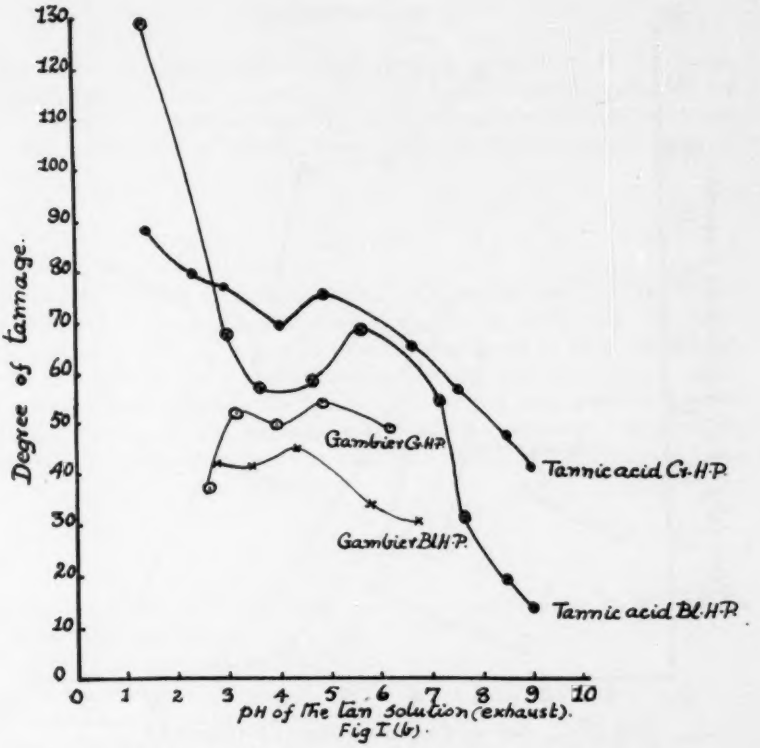

Fig I (b).

The higher the pH of the tan liquor, the lower is the acidity of the chromium sulfate left in the hide powder, which comes down to zero at high pH values. This is not the case with any of the ordinary neutralising agents in the same time, [9] showing the readiness with which the content of vegetable tan liquors, at least some, enter the chrome complex displacing the sulfate from chrome tanned leather. However, this is not of great importance to tanners, as such high pH values are not encountered in tanning practice. The effect of pH on the acidity is shown graphically in Fig. II.

Experiments with nontans from gambier throw interesting light on the way they are able to react with chrome tanned hide powder, displacing sulfate from the same. The ash of the solubles did not decrease much after the treatment, indicating the inorganic constituents of the tanning material to be of minor importance in displacing the sulfate from chrome tanned hide powder. So, most of the matter fixed by chromed hide powder from gambier nontannins consists of organic constituents, which were shown to enter the

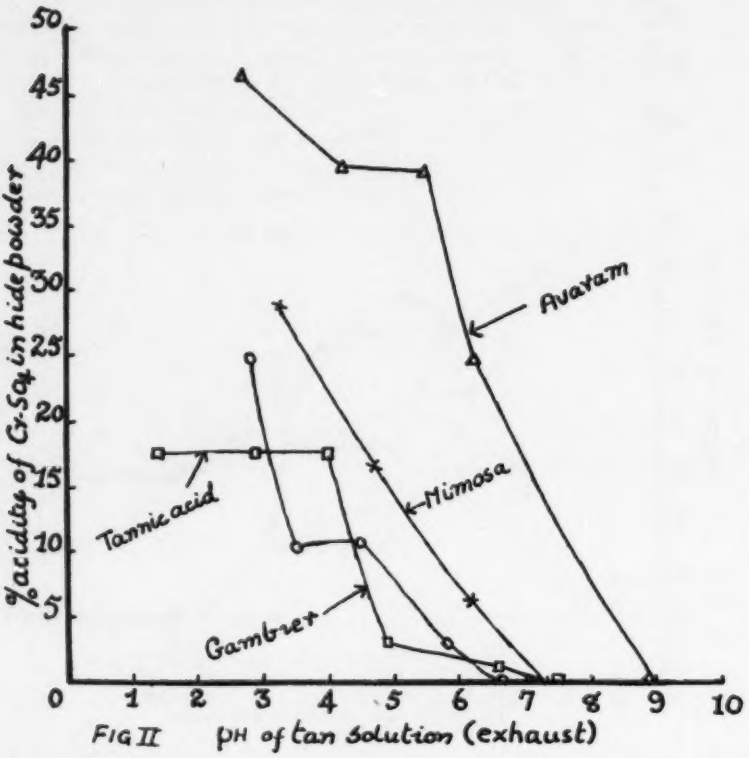

FIG II

chrome comlex in chrome tanned colagen.[10, 11]. Otto[10] in his interesting researches with gambier extract, has found 23 per cent anionic material in it by weight. On analysis of the anionic portion, one fourth was reckoned to be aromatic. He suggests that both of the acids identified were acids with molecular weight of about 150, in the anionic portion, the aliphatic being adipic acid and the aromatic one 2, 4 dihydroxy benzoic acid. Dihydroxy benzoic acid alone has twice the effect of gambier non tannins in reducing certain dye fixation on chrome leather and also increasing the penetration of mimosa extract into the same.

The trials with pieces of chrome tanned leather using Avaram along with other materials for comparison, open up the possibilities of combination tannages being used not only for army uppers[12], but also for various other types of tanning, where time could be saved and modern methods could be introduced, in the Indian leather industry.

Acknowledgments

The author expresses his sincere thanks and esteem to Dr. K. H. Gustavson, Director, Swedish tanning research institute, for his valuble suggestions and kind interest in the present work. His thanks are also due to the Government of India and the Statens Tekniska Forskningsrad for their grants for the same.

Summary

Vegetable tannage of chrome tanned hide powder and pelt is studied. Tannin fixation is not appreciably pH dependent as in the case of ordinary hide powder or pelt. The non tannins also are shown to have considerable effect in displacing the complex bound sulfate and thus reducing the acidity of the chromium sulfate in the leather. As the pH is increased the acidity of the chromium sulfate decreases and finally becomes zero. Experiments with calf skin pieces show that this particular process has many uses in India.

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Received May 26, 1954.

Histological Study of the Hog Skin Dehairing Process*†

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I. INTRODUCTION

The development of improved machine for defatting and dehairing hogskin sides by personnel of the Wolverine Shoe & Tanning Corporation, Rockford, Michigan, has created a potential source of raw material for production of 500,000,000 square feet of leather suitable for shoe uppers and other types of leather products [5]. In view of the constantly decreasing volume of horse hides, the importance of a suitable raw material to take its place is evident. Leather produced from hog skins of *satisfactory* quality is finding increasing use in this field. However, hog-skins of "satisfactory quality" are not available in sufficient quantity to meet even present day requirements, much less future ones.

Realization of this situation prompted a study to develop optimum conditions in the so called "scalding process" for hog carcasses whereby skins of inferior quality for leather purposes would be reduced to a minimum.

No studies have been made to determine the effects of time and temperature of the scalding bath on the tissue components present in hog skins such as epidermis, hair root, collagen and elastin fibers, etc., through application of histological techniques. The results reported herein have been obtained exclusively by this means.

Considerable attention has been given to this problem by Hausam [3] and by Stather [8], [9] in Germany. Our findings support the contention that temperatures above 140°F. for periods in excess of 6 minutes are undesirable for production of highest quality raw material. Neither of these investigators, however, specifically mentions a seasonal factor which is obvious in the slaughter cycle of domestic hogs and requires modification of the scalding conditions from season to season where equipment is of limited capacity.

II. MATERIALS AND METHODS

All samples used in this study were obtained from 200-240 lb. carcasses within half an hour after slaughter at Agar Packing and Provision Corporation, Chicago. They ranged in size from 2-15 square inches, and much of the attached subcutaneous fat was trimmed before they were fixed in 10 per cent neutral formalin for at least 48 hours before further histological processing.

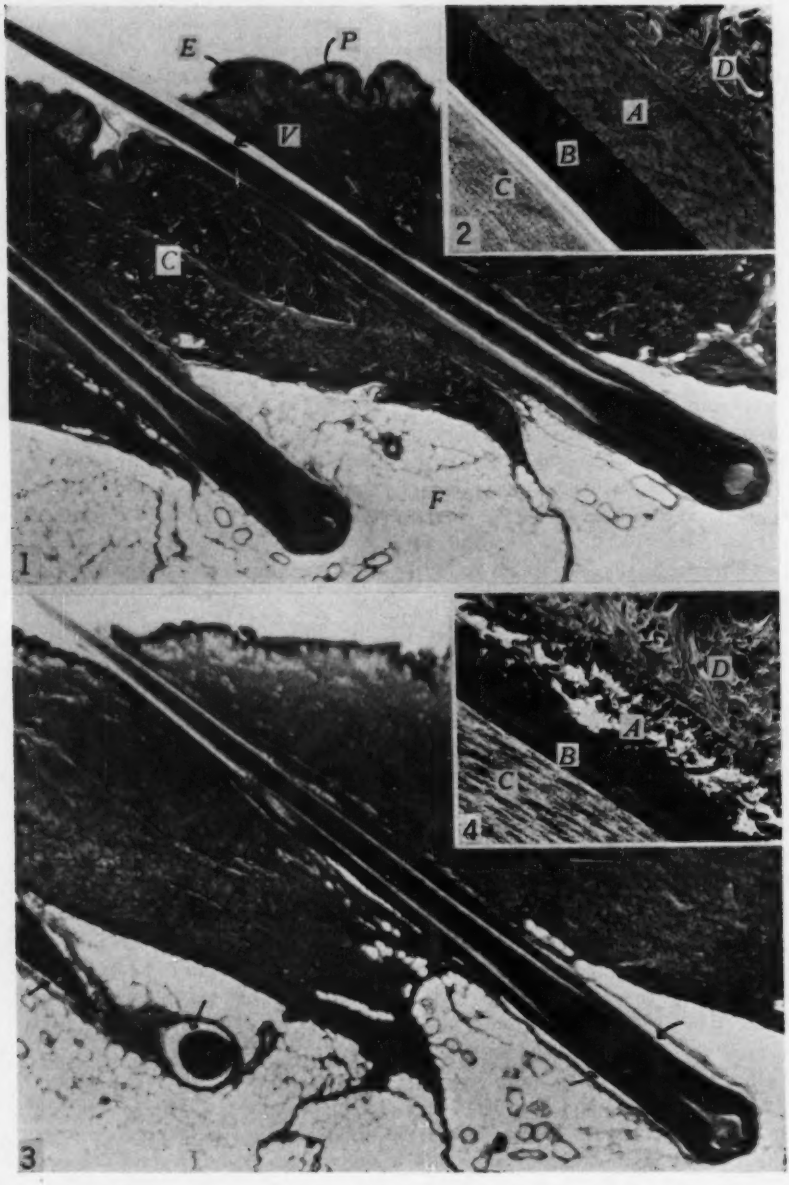
†This paper was presented before the 50th Annual Meeting of the American Leather Chemists Association, Bedford, Penna., June 6, 1954.

*Journal paper No. 97, American Meat Institute Foundation.

Five sets of samples were studied. The first set of 6 samples was taken from a single carcass during September: (1) after "bleeding" following slaughter (normal control); (2) after cold-water dip; (3) after scalding at 140°F. for 4 minutes; (4) after machine dehairing; (5) after singeing; and (6) after shaving. The second set was taken in October: (1) after cold-water dip (as normal control since it was found that this treatment caused no structural change whatsoever); (2) after scalding at 140°F. for 4 minutes; (3) after machine dehairing; (4) after normal singeing; (5) after excessive singeing (several times the normal duration); (6) after normal shaving; and (7) after excessive shaving. The third set was taken in November to study the effect of the time of scalding treatment. The samples were taken from individual carcasses which had been in the 140°F. scalding vat for 1, 2, 4, 6, 8, and 10 minutes, respectively. The fourth set, 8 samples, was taken in February: (1) 1-minute scalded; (2) 2-minute scalded; (3) 4-minute scalded; (4) 4-minute scalded and machine dehaired; (5) 8-minute scalded; (6) 8-minute scalded and machine dehaired; (7) 10-minute scalded; and (8) 10 minute scalded and machine dehaired—all at a scalding temperature of 140°F. The fifth and last set of samples was taken in April to study the effect of scalding at lower temperature levels: (1) scalded at 132°F. for 6 minutes; (2) scalded at 132°F. for 10 minutes; (3) scalded at 132°F. for 6 minutes and machine dehaired; (4) scalded at 132°F. for 10 minutes and machine dehaired; (5) scalded at 136°F. for 6 minutes: (6) scalded at 136°F. for 10 minutes; (7) scalded at 136°F. for 6 minutes and machine dehaired; and (8) scalded at 136°F. for 10 minutes and machine dehaired.

Blocks about ½ inch long and ¼ inch thick were removed from the formalin-fixed samples for histological processing. Some of the very early samples were studied in paraffin sections, but the paraffin technique was soon abandoned chiefly on account of the heat employed by the process. After that, standard frozen and celloidin sections were made from all materials. 20-micron longitudinal (vertical) sections were prepared by the freezing technique while the celloidin technique was used for both longitudinal and transverse sections.

Difficulties were encountered in making frozen sections due to the presence of abundant subcutaneous fat and, more particularly, to the fact that the hair roots were embedded in the fat. A special technique had to be developed to overcome these difficulties. The tissue blocks were washed in running water for 10-20 minutes, followed immediately by four changes of fresh acetone for a total of 4 hours to dissolve the fat. The defatted tissue was washed again in running water for 10-20 minutes and then placed in a mixture of Carbowax 1500 and distilled water (1:1 by weight) [4]. The function of the Carbowax was to give the tissue the necessary consistency and firmness which water alone could not provide. Usually a few hours in the Carbowax mixture would suffice before mounting the tissue on to the freezing micro-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tome. If, however, the material failed to cut satisfactorily, washing the block again in distilled water for 10 minutes or so facilitated proper freezing of the tissue.

Problems of another nature were experienced in preparing celloidin sections. For our purpose an ideal section would be one which showed the entire follicle lengthwise including the hair shaft, i. e., extending from bottom to mouth of a hair follicle (Figures 1 and 3, for example). In such a section, however, artifacts resulting from shrinkage at the follicle mouth were easily produced. This difficulty was solved by cutting the sections at slightly more than 10 microns thick, and instead of dissolving the celloidin, the celloidin section was stained *in toto*. In this way the celloidin served as a medium to prevent the occurrence of any alteration in the structural organization of the tissue.

A number of stains were used for the material. The frozen sections were studied either completely unstained or with Van Geison's alone for the study of collagen fibers. The celloidin sections were regularly stained with Pollak's trichrome [6], but sometimes also with hematoxylin and Van Gieson's. Orcein and Orange G were used to stain elastic fibers in those sections designated for the study of elastic fibers only.

Many of the significant results were recorded by photomicrography. Thirty-five mm. Kodachrome slides were made from the sections stained with Pollak's trichrome. Other photomicrographs were made on 4 x 5 inch black and white Kodak Super XX Panchromatic films from both stained celloidin sections and frozen sections for purposes of illustration.

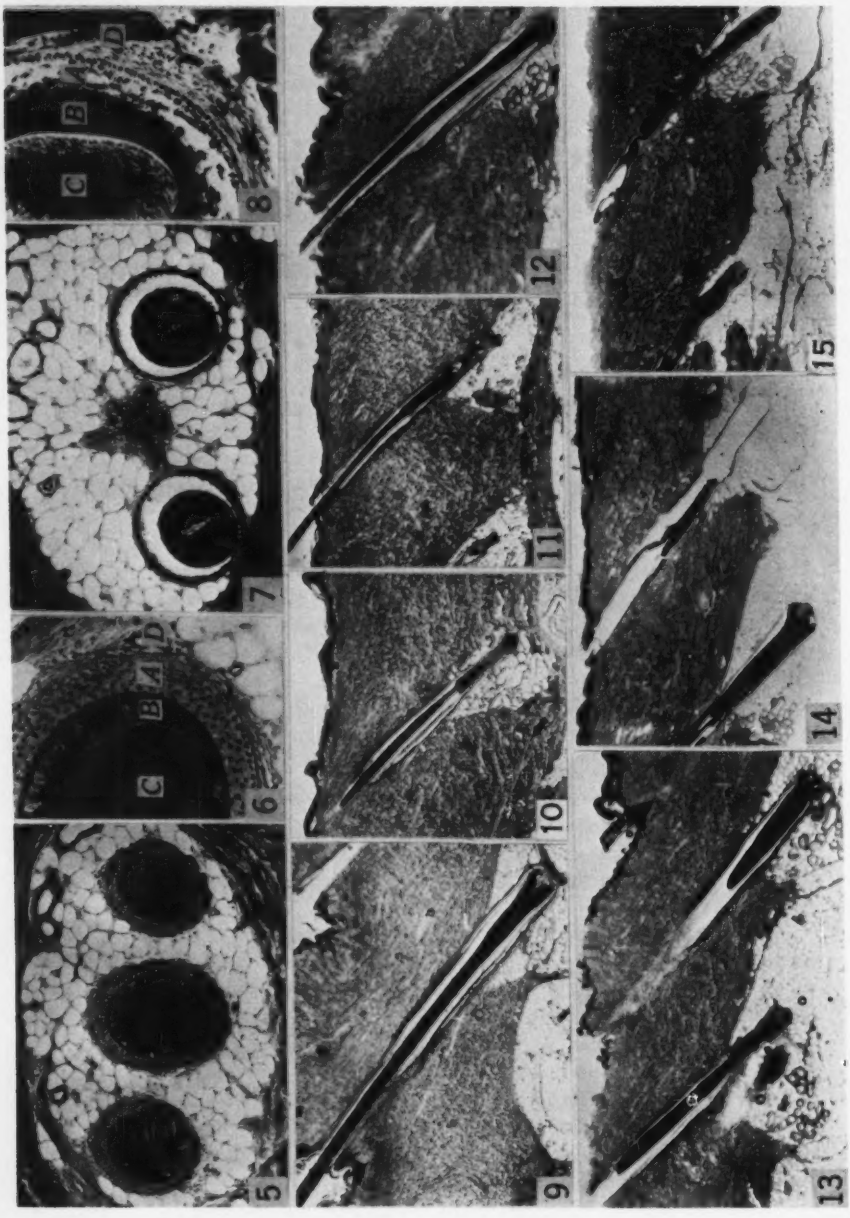
III. Results

A. *Normal Structure of Hog Skin.*

Hog skin has an epidermis (mostly *Stratum Malpighii*) approximately 0.1 mm. thick, with its lower boundary bordering on the dermal papillae (Figure 1, P). It is neither smooth nor of uniform thickness but ruffled with numerous folds and ridges. External to the epidermis and closely adhered to it are layers of cornified (dead) cells commonly known as the "scurf" (*Stratum corneum*).

The epidermis extends into the hair follicles, providing a complete lining for the latter. The hair itself is formed by cells at the bottom of the follicle, which proliferate at a high rate during the period of hair growth. A hair follicle measures approximately 5mm. in length from bottom to mouth. It is not vertical but inclined 40°–45° from the skin surface.

About the lower one-third of the follicle is anchored in adipose tissue (subcutaneous fat) underneath the dermal portion of the skin (*Corium*). The latter is a thick layer of connective tissue fibers, principally of the collagenous type. The *Corium* of cattle hide is, by comparison, much thicker and consequently the entire follicle is embedded in it [10].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The hair may be divided into hair sheaths and shaft. The former consists of two layers: (a) an outer hair sheath (Figure 2, A) which is continuous with the *Malpighii* layer at all times with decreasing thickness toward the bottom of the follicle; and (b) an inner hair sheath (Figure 2,B) which is thick near the base, becoming increasingly thinner higher up. These sheaths line about the lower two-thirds of the follicle and then terminate abruptly at a level where the sebaceous glands open into the follicular cavity (Figure 1,V). From this point upward, the hair is completely bare, i.e., deprived of a sheath. It consists of cells making up the hair shaft. The hair is held most firmly at its root. In fact, as can be readily seen in Figure 1, the basal portion of the hair is distinctly thicker than the average diameter of the follicle (see Discussion). In this region (about one-fourth of total follicle length) the hair root is tightly bound to the follicular wall. Above that, the follicular cavity separates the hair shaft from the sheaths or follicular wall.

B. *Structural alterations following scalding at* 140°*F. for* 4 *minutes.*

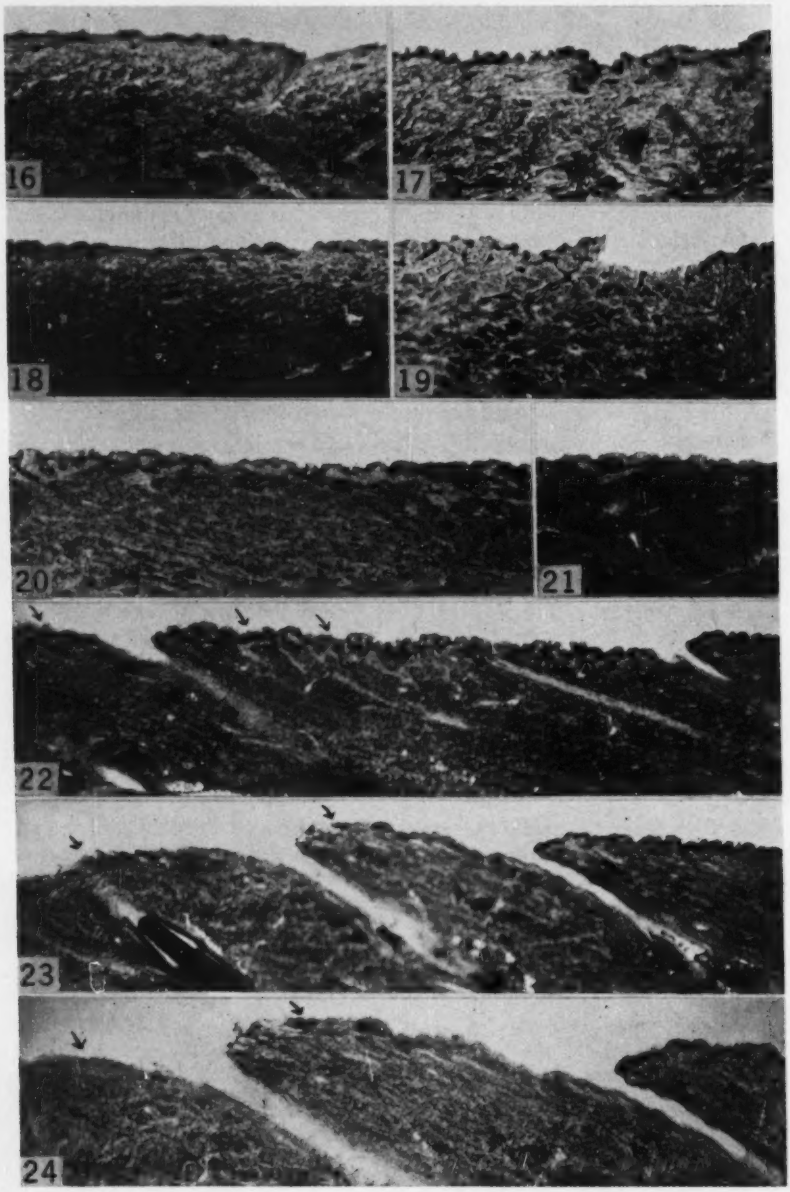
The observations described below are based on samples taken during September and October.

Effect of cold water dip on the skin structure. No appreciable changes in the structure of the entire skin were observed in the samples taken after the carcass was immersed in cold water prior to entering the scalding vat.

Effect of scalding on the skin structure (Figures 3 and 4). Scalding removed the scurf completely and occasionally softened the epidermis to the degree of stripping it either completely or for part of its thickness. However, this did not occur extensively; the skin epidermis remained intact for the most part.

A far more significant manifestation of scalding was found in the hair follicles in the form of a split within the layer of outer hair sheath (arrows, Figure 3). As a result of this cleavage, the cells of the outer hair sheath were divided into two layers (Figure 4,A). In some instances the new cavity so induced in the outer hair sheath extended all the way from the follicle base up to the point where the inner and outer hair sheaths merge. These changes were reflected in another unique observation. During sectioning of scalded skin on the freezing microtome many hairs dropped out of their follicles, and these hairs were found invariably to carry with them the entire inner hair sheath and the inner portion of the split outer hair sheath. This never happened to frozen sections of unscalded skin. Furthermore, hairs pulled out by force from unscalded skin do not carry the sheaths with them. These observations seem to reflect the structural changes occurring in the follicles during scalding, and the two phenomena together offer evidence as to what actually happens to the hairs during the mechanical dehairing process.

Effect of scalding on the structure of hog skin after dehairing (Figures 20 and 21). Dehairing completely removed the epidermis and any residual scurf. There were indications of occasional spotty damage to the surface layer of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the Corium which probably resulted from excessive local mechanical action. Typical examples of dehaired hog skin are illustrated by Figures 20 and 21.

In both of these examples, the uniformity and continuity of the homogeneous collagen layer is a noteworthy feature. This pattern, of course, makes the characteristic grain of the finished product. The normal grain pattern as shown does not have a flat surface. The reason for this is strictly anatomical, namely, specifically it is due to the presence of the dermal papillae (Figure 1, P).

Most of the follicles were empty or contained portions of the inner hair sheath, but never any outer hair sheath. Such an empty hair follicle was totally or partially collapsed. In the case of a follicle not successfully dehaired during the process, the hair usually exhibited signs of maceration, particularly at the root and lower portion. There were instances in which the hair was dislodged and disintegrated to a point beyond recognition, leaving behind only a mass of debris.

Effect of Excessive Singeing. Figure 16 represents the structure of hog skin after normal singeing. No serious damage of any kind could be detected although the character of the grain layer had obviously been somewhat altered (cf. Figure 20). The structures resulting from excessive singeing offered a totally different picture. As shown in Figure 17, excessive singeing had virtually nodulated the collagen, destroying the surface, thereby ruining the grain of any leather subsequently produced.

Effect of Excessive Shaving. Figures 18 represents the structure after normal shaving of a normally singed skin. The similarity in appearance between Figures 18 and 20 indicates that shaving, if done carefully, may not harm the grain. But tremendous damage can result when shaving is done excessively (Figure 19) on skin which has been normally dehaired and singed. The extent and character of the damage thus produced are certainly self-evident. The top of the collagen layer in this particular case appeared deeply nicked. It is readily apparent what this kind of damage would mean to leather quality.

C. *Effect of Scalding Time and Seasonal Variation.*

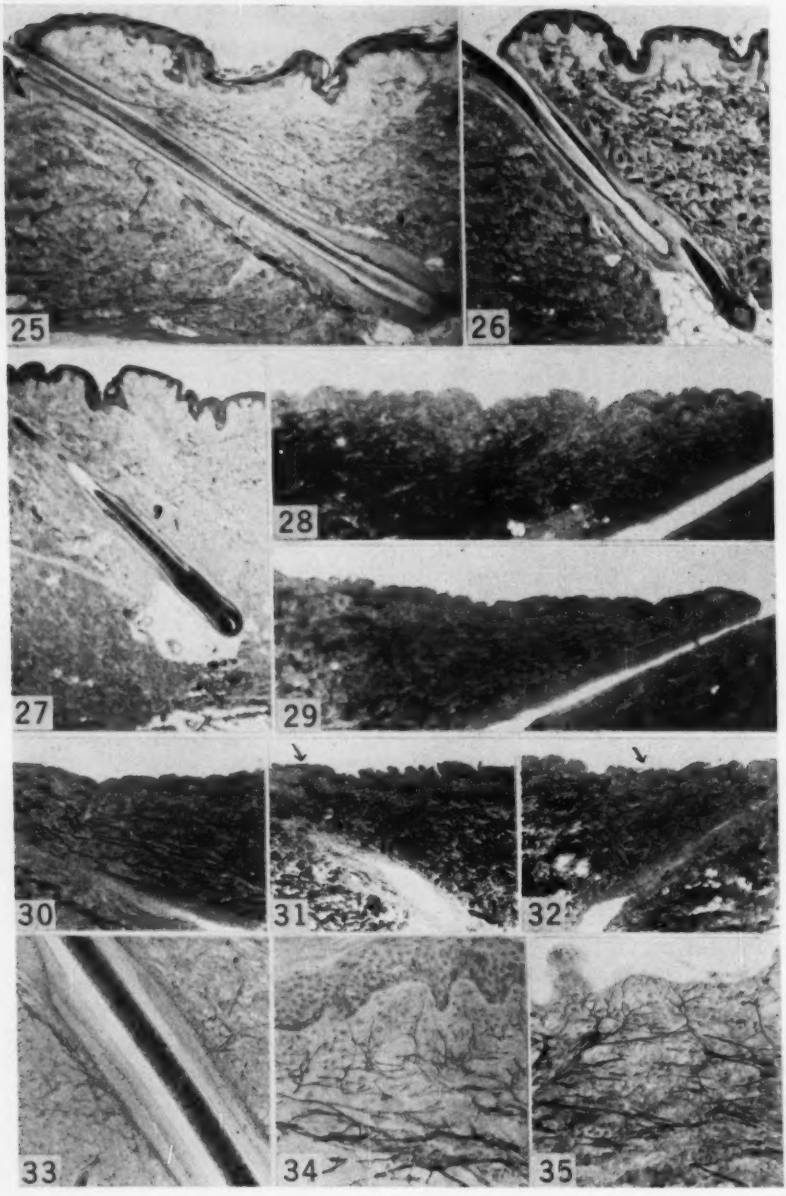
The following results are based on November and February samples scalded at 140°F.

1-minute scalding

No appreciable changes were found in either November or February samples scalded for 1 minute. The hair follicles and other skin structures were almost indistinguishable from those of unscalded (normal) hog skin.

2-minute scalding

Follicles in the 2-minute scalded November sample seldom, if ever, revealed any changes in the skin structure attributable to the scalding treatment.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On the other hand, the corresponding February sample showed many follicles which already had a noticeable cleavage in the outer hair sheath (Figure 10).

4-minute scalding

The November sample confirmed the results described under Section B. Most of its hair follicles showed a wide cleavage in the outer hair sheath (Figures 4 and 8). Sometimes such a cleavage may involve the whole circumference of the follicle and extend to a level where the two hair sheaths merge. It was further observed that the cells of the outer hair sheath where the cleavage had taken place were darkly stained (cf. Figures 4 and 8 with Figures 2 and 6, respectively) owing to the fact that these cells contained a large amount of glycogen [1]. The scalding, however, did not affect the epidermis.

Most follicles in the February sample scalded for 4 minutes showed a very extensive split in the outer hair sheath, generally more pronounced than that seen in the corresponding November sample. Frequently, portions of the epidermis were sloughed (Figure 12). However, after dehairing, the skin appeared to be indistinguishable from similarly treated samples taken in September, October, and November. The grain of these samples can best be represented by Figures 20 and 21.

6-minute scalding

No significant differences of any kind were noted between the November samples scalded for 4 minutes and for 6 minutes. Thus, based on the structures of the hair follicles and barring individual variations, 4 to 6 minutes' scalding at 140°F. may constitute a workable range for production of hog skins for tannery purposes.

8-minute scalding

Indications of advanced scalding effect were observed throughout the November sample in addition to cleavages occurring in the outer hair sheaths. For example, two follicles are shown in Figure 11. In one of them the hair had been detached from its root and pushed up for a distance of nearly 1 mm. The other follicle (only its basal portion shown on the left) represented another manifestation of severe effect from scalding: disintegration of the hair root. In other cases hairs were seen detached from follicles and they may have been cast out of the follicle completely. Since this happened during scalding, the phenomenon may be termed "premature dehairing."

These effects can be seen even more clearly in transverse sections (Figures 7 and 8) and, for contrast, compare them with Figures 5 and 6. Note (Figure 7) the wide cleavage in the outer hair sheath in two of the regular "triad" while the middle follicle has collapsed, showing some debris instead of hair.

Conceivably, the section cut through a level such as represented by the left-side follicle in Figure 11 where the hair had been dislodged and disintegrated.

Again, a greater scalding effect resulted in a similarly treated sample taken in February. Over-scalding was indicated by various degrees of disintegration of hair in addition to the usual cleavage of the outer hair sheaths which, in this sample, extended almost the entire follicle length. Note also that the sample represented by Figure 13 had some of its epidermis stripped during scalding.

The grain of this sample after dehairing (Figure 22) was, by preliminary examination, not different from that of skins conventionally scalded (i. e., 4 minutes), as illustrated by Figures 20 and 21. Upon more critical examination, however, fraying and roughening of the terminal collagen which would likely result in coarser and weak-grained leather, were indicated (arrows, Figure 22).

10-*minute scalding*

Results from the November sample were, by and large, similar to those scalded for 8 minutes except for a noticeably increased degree of epidermis stripping. Gross observations of pieces of this skin from the dehaired carcass, with the aid of a binocular dissecting microscope, revealed many broken ends of collagen fibers. The damaged condition of the grain was thus detected.

On the other hand, much new evidence of over-scalding was obtained from the 10-minute scalded February sample. It had many empty follicles indicating that hairs had dropped out during the process of scalding (right, Figure 14). Furthermore, follicles that had retained hair showed evidence of excessive structural changes, e. g., broken hair root together with marked cleavages in outer hair sheaths such as portrayed by another follicle in Figure 14 (left). These are regarded as signs of a greater scalding effect than seen in the similarly scalded November sample.

Of even greater interest was the finding that, after dehairing, the 10-minute scalded sample was found to have retained hair in many of its follicles (Figure 15). In such cases, the hairs were so deformed and disintegrated that it was impossible to remove them. It is also likely under these circumstances that the usual evidence of scalding (i. e., cleavage of the outer hair sheath) may have become masked.

Evidence of damaged grain layer in this sample was consistently observed. (arrows, Figures 23 and 24). Portions of the surface collagen were actually missing. Observations made on pieces of skin taken from the carcass as it emerged from the dehairing machine indicated that some of the collagen fibers may have been stuck to the epidermis and thus were torn off with the epidermis during dehairing.

Elastic Fibers in Hog Skin

Many preparations both after scalding and after dehairing were stained specifically for elastic fibers in an effort to determine whether or not the elastic fiber network surrounding the hair follicles may be involved in any way with the cleavage of the outer hair sheath already established as a result of scalding and whether or not excessive scalding followed by dehairing might adversely affect these fibers to the extent of influencing leather quality.

The elastic fibers of the hog skin consisted of a rather fine network distributed more or less uniformly throughout the collagen layer (*Corium*). Slight concentration of these fibers was noted at two places: surrounding each hair follicle (Figure 33), and underneath the epidermis (Figure 34). These fibers, which stained a dark blue by orcein against a background of yellow collagen stained by orange G, branched profusely and steadily decreased in diameter. The finest branches are represented by those terminating underneath the epidermis (Figures 34 and 35). The very tips of some of these fine fibers measured less than 0.1 micron in thickness.

No changes in the elastic fiber system that could be attributed to either scalding or dehairing were observed. After 8 minutes of scalding and even after dehairing of a 10-minute scalded skin (Figure 35) the delicate network of elastic fibers remained intact and unaltered from that seen in the conventionally scalded sample (Figure 34). Similarly, no changes were observed in the elastic fiber network surrounding hair follicles in scalded or dehaired samples (Figure 33).

D. *Effect of Lowering Scalding Temperature.*

Results from the group of carcasses scalded at lowered temperatures and taken in the month of April were clear cut and definitely in favor of employing a scalding temperature 4 to 8 degrees lower than 140°F. insofar as ultimate leather quality is concerned.

Well Dehaired Carcasses. The carcasses scalded at 132° or 136°F. emerged from the dehairing machine suprisingly well dehaired. They were much more free of hair than most of the carcasses scalded during the "hard hair" season (September, October, and November) at 140°F. for 4 minutes. Of the four carcasses scalded at these two lowered temperatures, the one scalded at 132°F. for 6 minutes had by comparison a few more unremoved hairs than the other three.

Unchanged Structures of the Hair Follicles. None of the carcasses scalded at these two temperatures for 6 or 10 minutes showed any sign of structural alteration of the skin. Both the epidermis and hair systems were intact, i. e., there was no cleavage in the outer hair sheath such as was found consistently in skin scalded at the conventional temperature of 140°F. Figures 25, 26,

and 27 represented three different stages in the growth cycle of a hair, yet in every case both the hair and follicle were structurally intact. In the face of these findings, it may be concluded that the occurrence of a cleavage in the outer hair sheath is not a required condition for successful dehairing. But if such a split does occur, it probably facilitates dehairing.

Acceptable Grain Layer of Dehaired Skin. It was of interest to find that none of the scalding conditions under discussion (even 136°F. for 10 minutes) harmed the grain layer to any appreciable degree (Figures 28, 29, and 30) It is to be noted that in all these cases dehairing was successful as evidenced by the empty follicles shown in these figures. The grain layer in all of them appeared normal. Areas with somewhat damaged grain were detected occasionally (arrows, Figures 31 and 32). These instances, however, were of infrequent occurrence, and therefore not considered to be of great importance.

IV. Discussion

Certain indications from this work may be considered of practical significance. First of all, 140°F. is not a safe temperature for scalding because at that temperature damage to the grain layer is apt to result from scalding for 4 minutes, and certainly to occur if carried beyond 6 minutes. Second, the practice of singeing and shaving presents the potential danger of inflicting substantial damage to the grain in skins which would have otherwise been acceptable. Third, at least during the "easy hair" season, 132°-136°F. constitutes an effective scalding temperature range, which is essentially harmless to the grain for as long as 10 minutes of scalding, and yet produces carcasses that can be cleanly dehaired. It need only to be ascertained how long the carcasses should be scalded at this low temperature during the "hard hair" season. But the results clearly point to the use of relatively low scalding temperatures to secure skins capable of producing high quality leather.

Scalding has been an age-long practice not only for hogs but for poultry as well[2]. Recently, hog scalding has been closely scrutinized and discussed with somewhat conflicting views[3, 8, 9]. Our results, while not completely agreeing with Stather that scalding should be condemned and abandoned altogether, nevertheless substantiate on a structural basis most of the claims made about hogskin damage resulting from the process. It is evident from our results as well as from operating experience that scalding intensity depends on both temperature and time. Therefore, optimum scalding, one which yields the best possible leather and yet dehairs without trouble, can be obtained only by a right combination of these two factors. Unfortunately, because of the complication arising from seasonal variation, the results to date do not permit a decision as to what constitutes optimum scalding. However,

the results do suggest the advantage of employing relatively low temperatures for scalding since hog skin can withstand scalding for much longer time at lower temperatures than is possible at higher temperatures. While 4-6 minutes is not considered a safe range for scalding at 140°F., this range can be easily extended to 10 minutes at 132–136°F., possibly much longer for 132°F. Needless to say, such an extended safety factor is much desired.

The results also have demonstrated certain signs of over-scalding, which might serve as warning for approaching the danger point. While epidermis sloughing and stripping and premature loss of hair occurred during the process of scalding, the true danger signal was the retention of disintegrated hairs by follicles in dehaired carcasses. For when that happens, grain damage is almost certain to occur. Both the retained hair and the accompanying damaged grain are, of course, undesirable, hence to be avoided whenever possible. In this connection, since hair follicles without a split in the outer hair sheath nevertheless could properly dehair, the occurrence of the split might be also considered as exceeding the danger point.

A possible explanation for the seasonal factor on the ease of dehairing may relate to a response of the animal to climatic conditions. During the "hard hair" season (September, October, and November) most of the hairs are in active growth period when the hair shafts are held most tightly within the follicle, hence difficult to remove. This is in preparation for the coming winter months when a thick fur is in demand to keep the animal warm. In the spring, with oncoming of warm weather, most of the hairs have fully grown or reached a resting stage or, in some instances, a new hair has commenced to regenerate below an old one (see Figures 25 and 26[1]). These seem to be the conditions making for the "easy hair" season. Physically speaking, too, these old hairs are held much less tightly than the actively growing ones encountered in the winter.

A sound understanding of the mechanism of scalding may logically proceed from a structural basis such as just presented. During the "hard hair" season while the hairs are actively growing, the base of hair (bulb and root, about $\frac{1}{4}$ of the follicle length) is actually thicker than the diameter of the follicle (see Figure 1); the rest of the hair shaft is closely adhered to the follicular wall. These are some of the anatomical reasons for difficulties encountered in dehairing during the winter months. It is postulated that under these circumstances scalding causes an increase or widening of the follicle diameter at the critical temperature (much more important than scalding time[7]) of 140°F. at which collagen begins to lose moisture and shrink. The widened follicle so induced was invariably accompanied by splitting of the outer hair sheath when carcasses were scalded at this temperature during the "hard hair" season, but not at 132° or 136°F. in the "easy hair" season. Presumably, therefore, collagen shrinkage either did not take place at all

or not to the extent of causing the outer hair sheath to cleave at these lower scalding temperatures. Dehairing was achieved apparently without the effect of scalding, but rather by virtue of structural character of the follicles as above mentioned.

It is obvious from the above discussions that unanimity regarding the optimum conditions for scalding of hog carcasses is, as yet, lacking. Further studies of the nature reported here are essential both from the standpoint of understanding the "seasonal" factor and the effect on the quality of subsequent leather produced therefrom. It will be of importance to continue such studies during the "hard hair" season.

V. Summary and Conclusion

This study was undertaken with the ultimate objective of improving quality of hog skins for tannery use.

Fresh skin samples from variously treated hog carcasses were fixed in 10 per cent neutral formalin and processed with modified techniques for frozen and celloidin sections. The following results, histologically evaluated, have been obtained from these samples.

Hog carcasses scalded at 140°F. for 4 to 6 minutes had, with few exceptions, intact epidermis. A cleavage within the outer hair sheath split it into two layers which, however, did not result generally in the loss of hair. When machine-dehaired, the scalded carcass lost all epidermis and most hair, thereby exposing the collagen surface (grain), which was in most cases undamaged. The next two processes which are currently practiced following dehairing, namely, singeing and shaving, could damage the grain as was evidenced by the fact that samples taken from parts of a carcass intentionally subjected to excessive singeing or shaving revealed substantial grain damage.

Evidence of over-scalding was obtained from samples scalded for 8 and 10 minutes at 140°F. These consisted of sloughing of epidermis and various manifestations of an enhanced effect on the follicle, e. g., wider and more extensive cleavage in the outer hair sheath, pushing up of the bulb and disintegration of hair shaft, often resulting in loss of hairs during scalding. Such a carcass usually emerged from the dehairing process with numerous hairs wholly or partially unremoved and with the grain layer visibly damaged. The retained hairs in the skin were found to be disintegrated to a point that they could not possibly be withdrawn from their follicle. These results clearly indicate the undesirability of over-scalding not only insofar as leather quality is concerned, but even from a meat processing standpoint.

EXPLANATION OF FIGURES

Fig. No.	When Taken	Treatment	Scalding temperature	Scalding time (min.)	Sections	Stains used	Mag. (x)	Features shown (further explanation in text)
1	Sept.	None			16 μ celloid.	Pollak's trich.	26	Normal structure of hog skin: Epidermis (E), Corium (C), Fat (F), dermal papillae (P), follicular cavity (V).
2	Nov.	None			"	"	165	Vertical longitudinal section of normal hair follicle: outer hair sheath (A), inner hair sheath (B), hair shaft (C), corium (D).
3	Sept.	scalded	140° F	4	"	"	26	Cleavage (arrows) in outer hair sheath of two follicles, one cut obliquely.
4	Nov.	"	"	4	"	"	165	Enlarged view of split outer hair sheath (A) labeled as in Fig. 2.
5	"	"	"	1	"	"	36	Typical arrangement of hair follicles.
6	"	"	"	1	"	"	100	Enlarged view of intact hair follicle labeled as in Figs. 2 & 4.
7	"	"	"	8	"	"	36	Collapsed middle follicle and wide cleavage in outer hair sheath in remaining 2 follicles.
8	"	"	"	4	"	"	100	Enlarged view of split outer hair sheath in scaled follicle labeled as in Figs, 2, 4, 6.
9	"	"	"	10	20 μ frozen	None	10	Split of outer hair sheath
10	Feb.	"	"	2	"	"	10	Split of outer hair sheath.
11	Nov.	"	"	8	"	"	10	Pushing up of hair shaft.
12	Feb.	"	"	4	"	"	10	Epidermis sloughing, cleavage in outer hair sheath and disintegration of hair root.
13	"	"	"	8	"	"	10	Epidermis sloughing with damage to underlying collagen and severe scalding effect on follicles.
14	"	"	"	10	12 μ celloid.	Pollak's trich.	10	One empty follicle and another containing a shattered hair
15	"	"	"	10	"	"	10	Unsuccessful dehairing due to overscalding
16	Oct.	dehaired & normally singed	"	4	20 μ frozen	None	20	Slightly damaged grain layer
17	"	dehaired & excessively singed	"	4	"	None	20	Markedly damaged grain layer

Fig. No.	When Taken	Treatment	Scalding temperature	Scalding time (min.)	Sections	Stains use	Mag. (x)	Features shown (further explanation in text)
18	"	dehaired, singed & normally shaved	"	4	"	None	20	Slightly damaged grain layer
19	"	dehaired, singed & excessively shaved	"	4	"	None	20	Severely damaged grain layer
20	"	scalded & dehaired	"	4	"	None	20	Normal grain layer
21	Feb.	"	"	4	"	Van Gieson's	20	Normal grain layer
22	"	"	"	8	"	None	20	Slightly damaged grain layer (arrows)
23	"	"	"	10	"	"	20	Markedly damaged grain layer (arrows)
24	"	"	"	10	"	"	20	Markedly damaged grain layer (arrows)
25	April	scalded	132°F.	10	14 μ celloid.	Pollak's trich.	14	Unaltered structure of a resting follicle
26	"	"	"	6	"	"	14	Unaltered structure of an old follicle and a new regenerating hair.
27	"	"	"	6	"	"	14	Unaltered structure of an active follicle.
28	"	scalded & dehaired	136°F.	10	"	"	20	Nearly normal grain layer
29	"	"	"	6	"	"	20	Nearly normal grain layer
30	"	"	"	6	"	"	20	Nearly normal grain layer
31	"	"	"	6	"	"	20	Slightly damaged grain layer (arrow)
32	"	"	"	10	"	"	20	Slightly damaged grain layer (arrow)
33	Nov.	scalded	140°F.	4	"	Orange G and Orcein	32	Unaltered elastic fibers around hair follicle
34	"	"	"	4	"	"	156	Unaltered elastic fibers underneath epidermis
35	Feb.	scalded & dehaired	"	10	"	"	156	Unaltered elastic fibers after removal of epidermis

The influence of a seasonal factor upon the scalding efficiency was indicated. For instance, samples taken in the months of September, October, and November showed no structural changes after 1 and 2 minutes of scalding at 140°F., while samples taken in February showed that cleavage had been initiated in the outer hair sheath after only 2 minutes of scalding. Samples scalded for 4, 8, and 10 minutes in February revealed scalding effects greater than those manifested by samples scalded for these periods taken during the "hard hair" season months, September to November.

All samples taken in April, scalded at 132° or 136°F. for 6 or 10 minutes, had intact epidermis and hair follicles and exhibited no detectable effect of the scalding treatment. These carcasses, on the whole, dehaired cleanly by the usual machine dehairing process, and the dehaired skins revealed little or no damage to the grain layer. These results indicate that both temperature and time ranges used in these latter experiments are desirable and definitely superior to those generally in use. However, allowance should be made for the fact that these results were obtained during the "easy hair" season.

Implications of these findings to practical aspects of preparing hog skins for leather have been discussed.

Acknowledgments

We are indebted to the Wolverine Shoe and Tanning Corporation, Rockford, Michigan, for financial support of this study, and to the Agar Packing & Provision Corporation, Chicago, for the interest and cooperation of personnel in furnishing samples of hog skins used in these studies.

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Received, July 28, 1954.

Discussion

William T. Roddy: The presentation by Dr. DeBeukelaer has given us a very graphic demonstration of the structural changes which occur during the hog skin dehairing process. The conclusions which he has drawn fits the pattern observed by Stather in 1953 in regards to damage which occurs when the scaling process is prolonged.

Stather's findings indicate that scalded skins have ten to twenty-three per cent less strength than unscalded skins. He concluded that scalding blood-warm hogs for four to six minutes at 59 to 62 degrees Centigrade, even with careful operation, caused considerable damage, (which at times cannot be recognized externally) either on the raw hide after dehairing, or on the finished leather.

Now the histological techniques employed in this study show exactly what damage is being done, not only to the collagen on the raw hide but equally to the finished leather.

The use of hot rosin in some of the American packing plants for dehairing of hog hides probably results in as much or more damage than the scalding process and perhaps Dr. DeBeukelaer would like to briefly discuss that practice.

F. L. DeBeukelaer: Mr. Roddy, while I have had no direct experience with this rosin composition for dehairing, I do know from the Wolverine Shoe and Tanning Corporation, that they will not buy any skins that have been dehaired by this process because they cannot make satisfactory leather out of them.

I also know from my former experience in following gelatin production operations, of which pork skins are widely used as the raw material, that it produces an inferior gelatin both in color and in physical characteristics of quality, which are viscosity and jelly strength. So they are not used in edible gelatin manufacture.

The only place I know of where the rosin-dipped skin has any use outside of going to the tanks is possibly in the making of hide glue. But even there you have an inferior glue as measured by the physical constants that I mentioned, as compared to calfskin or cattle hide raw material.

Roddy: As we all observed in the cross-section of the fresh skin, it has the ideal structure. Now this structure, if it was shipped to the tannery, in let us say, the fresh state, and the tanner placed directly in the brine and tanned it into a leather, it would make the ideal leather as far as pig skin is concerned. I think at present a process like that is being carried on. I wonder if Dr. DeBeukelaer might have data comparing this particular type of stock to the stock which has been scalded.

DEBEUKELAER: That is frozen or salted?

RODDY: Yes.

DEBEUKELAER: I have had no particular experience because we have not carried our work beyond the laboratory bench scale operations. But I would anticipate from the nature of collagen and its behavior in brine solutions that you would get superior leather. If you were able to ship these skins either under refrigeration and unsalted, or by salting or curing, much as you cure brined hides, either form should produce a superior leather, I would say.

RODDY: It is also possible to unhair fresh calfskin or fresh steer hide by the scalding process, using the temperature of 59 to 62 Centigrade for five minutes. And while there has been no report of the study on these two stocks, it should apply equally well to either hide.

BOOK NOTICES

Polymer Degradation Mechanisms. Proceedings of a symposium, under the chairmanship of G. M. Kline. National Bureau of Standards Circular 525. Available from the Superintendent of Documents, Washington 25, D. C., 280 pages. buckram, $2.25. Seventeen papers by international contributors, reviewed, in 1951, the postulated mechanisms for the degration of such synthetic polymer systems as the vinyls, the acrylics, cellulose esters, polyethylene and the polyamides. The papers are frequently supplemented by extensive bibliography and a short discussion. The increasing use of resinous materials in leather finishes makes this book especially timely for those interested in the stability of leather finishes, especially upholstery finishes. The paper on polyamides, covering 27 pages, gives information on deterioration processes involving a peptide bond linked polymer which is particularly suggestive for those interested in collagen chemistry. The calibre of all the contributors to the volume assures its technical excellence; excellent book paper, a clear type font and a good binding combined to produce an outstanding volume.

R. M. L.

The Physical Chemistry of Dyeing and Tanning. A discussion of the Faraday Society, Sir Hugh Taylor, chairman. Aberdeen University Press Ltd., 6 Upper Kirkgate, Aberdeen, England, 1954, 256 pages, 35 shillings. The discussion is primarily concerned with the dyeing of fibers rather than with tanning. Twenty-three papers, supplemented by bibliography, and two general discussions comprise the volume. The British Leather Manufacturers Research Association has contributed a paper on diffusion processes in tanning, by D. M. G. Armstrong, and a paper on the interaction of tanning materials and collagen monolayers, by S. C. Ellis and K. G. A. Pankhurst. Other papers of particular interest to leather chemists are those by Schulman and Dogan on the reaction of chrome salts on fatty acid, amino acid and protein monolayers, a paper by Pouradier on the chrome alum hardening of gelatin, Gustavson's paper on multi-point fixation of chromium by collagen, and the paper on hide and skin mucoids, or inter-fibrillary material, by Burton and Reed. This volume is recommended to any leather chemist interested in theoretical aspects.

R. M. L.

ABSTRACTS

The Art of the Saddler. By M. de Buccar. *Rev. Tech. Inds. Cuir.* **43,** 92 (1951). A brief description of saddle-making as practiced in London and Walsall. The foundation is the saddle-bow which is made of beech wood and steel. Part of the art of the saddler consists in choosing the best types and selections of leather for the different parts of the saddle; part to his skill in trimming the pieces to fit; and part in laying out and stretching the leather so that each stitch is subject to the same strains in use and able to hold the padding in place for life. The saddler uses a sort of knee-vice to hold the work, and a crescent-shaped knife for trimming. Quality is maintained by the Honourable Corporation of Saddlers who, up until 1850, drew attention to, and punished, defective work by burning same in front of the offending saddlery. A. N. K.

Wetting and Penetrating Agents and Detergents. *Rev. Tech. Inds. Cuir.* **43,** 97 (1951). The increased use and number and types of these materials is discussed. Particular mention is made of an alkyl sulfate obtained from Scottish Shale and sold under the name of "Comprox". This product combines high wetting, penetrating and detergent properties. It is supplied in two forms: Type T is a soft white paste, of slightly alkaline reaction, specially prepared for the textile and bleaching industries. Type A is a clear amber liquid, of neutral reaction, miscible in all proportions with hot or cold water, stable over a wide range of pH, and does not form insoluble calcium or magnesium salts. According to the producers, Comprox can be used in the tannery: (a) as a soak-assist; for salted hides, 0.25% on the hide weight improves wetting back and removal of salt, blood and dirt; for sundried skins, 0.5% is suggested: (b) in chrome tannage, 0.5% speeds penetration of the chrome: (c) 0.5% on the hide weight added to the oil-mix improves penetration of the oil: (d) in dyeing, penetration and coloring are improved by 0.25% of the product. Comprox is not compatible with certain basic dyes, so it is well to test compatibility before use. It is compatible with other types of dye. A. N. K.

A New Wax for Humidity Protection. *Rev. Tech. Inds. Cuir.* **43,** 97 (1951). The usual practice of protecting electrical equipment, especially conduit, against humidity and short-circuits arising therefrom, has been to impregnate same with a varnish and then, in many cases, with a coating of microcrystalline wax. Results have been only moderately satisfactory where the equipment is in contact with water or detergents. Glyco Products Co., have produced Wax S-1167 which appears to be much more resistant under test. This product is a dark-colored synthetic organic wax, melting at 120°-125°C., and boiling above 250°C. It is tough and non-brittle, even at low temperatures. It has good electrical characteristics and resistance to water and oils. The molten wax adheres well to metal surfaces, with a minimum of shrinkage. A. N. K.

Vegetable Tannage by Means of Modified Collagens and Polyamides. K. H. Gustavson. *J. Soc. Leather Trades' Chemists,* **38,** 162 (1954). From considerations of the availability of reactive protein groups, the possible binding sites are: (a) Peptide bonds (keto-imide groups) which are not internally compensated (hydrogen bonded) by coordination of adjacent links, and hence are able to form hydrogen bonded structures by means of the CO- and NH- groups with the phenolic groups of the tannin. (b) Ionic groups, such as the ϵ-amino group of lysine, the guanidyl group of arginine and the carboxyl groups of the residues of glutamic and aspartic acids. (c) Hydroxy groups of residues of serine and hydroxyproline. (d) Amide groups of the NH_2CO- residues. A brief outline of the evidence supporting the view that the main binding group is the keto-

imide group, although not to the exclusion of the other groups, is given. Previous work by the author and a co-worker has given direct evidence that the peptide bond of collagen is available for tannin binding by showing that modified polyamides in the hydrated state combine vigorously with vegetable tannins. In this study the amounts of mimosa tannins and gallotannic acid irreversibly fixed by collagen and polyamide, as a function of the pH, were determined. Hide powder, hide powder in NaCl to prevent swelling, methylated hide powder and the polyamide were used. The polyamide posseses marked affinity for vegetable tannins, binding them irreversibly. The fixation of the condensed type of tannins (mimosa) by the polyamide is independent of the pH value of the system in the range 2-8. The same type of curve is shown by mimosa tannins in reactions with collagen, if swelling is prevented by salt addition. The results indicate the keto-imide groups to be the main binding sites of collagen for vegetable tannins of the type of mimosa tannins, and the phenolic groups to be the reactive sites of these tannins. Tannic acid, which carries a fairly strong acidic group, probably carboxylic as indicated by the titration curve, does not show this pH independence for its fixation of polyamide nor for its interaction with collagen at a constant degree of swelling. Other findings also indicate that, in the fixation of tannic acid by collagen, ionic protein groups are involved, besides the -CO•NH- groups which also function as binding sites for the gallotannins. From data on the fixation of vegetable tannins free from ionic groups, it is inferred that in the initial stage of vegetable tannage, the basic protein groups are mainly involved in the very rapid reaction of the fixation of tannins, whereas the secondary, slow fixation of the tanins is indicated to be a co-ordination of the tannins of the non ionic groups of collagen, probably the keto-imide groups.

R. H. T.

The Determination of Functional Groups in Tannins and Lignins. Part I. - Determination of Hydroxyl Groups. W. E. Hillis. *J. Soc. Leather Trades' Chemists,* **38,** 177 (1954). Hydroxyl groups have usually been determined by acetylation and purification until a constant acetyl content is obtained, and calculating from this the original hydroxyl content. This method is considered unsuitable. A method based upon acetylation with acetic anhydride and pyridine, which had been previously studied, was again critically examined. Acetylation was carried out for several days at room temperature, the excess aetic anhydride hydrolyzed with cold water and the acid determined electrometrically. The procedure is given in detail. Materials tested were pyrogallol, phloroglucinol, catechin specially purified, vanillin three time crystallized eugenol distilled under vacuum trimethoxy benzene made from pyrogallol, and aromadendrin (3, 4, 5, 7-tetrahydroxy flavanone). In all cases the determined hydroxyl content was very close to the theoretical. For example the measured value was 99.7 per cent for pyrogallol, 99 per cent for phloroglucinol, 98.8 per cent for eugenol, 98 per cent for catechin and 100.4 for aromadendrin. Vanillin gave variable results. Various lignin fractions gave consistent results.

R. H. T.

The Influence of Fatliquor on Spue Formation and Run in Chrome Gloving Leather: The Effect of Variations in Processing and Storage Conditions. R. H. Hall, R. G. Mitton and K. G. A. Pankhurst. *J. Soc. Leather Trades' Chemists,* **38,** 183 (1954). Previous work has established that run and tendency to spue are dependent upon the choice of fatliquor: but no correlation was found between the amount of spue and the analyses of the fatliquors used. Furthermore different tanneries did not give the same amount of spue with the same fatliquor, indicating that other factors than kind of fatliquor are involved. Hence a study of tanning process was indicated. Since the process subsequent to tanning appeared more likely to influence spue formation, factors after tanning were se-

lected. The following table gives the factors and the level at which they were investigated.

TABLE

Factor	Low Level	High Level
pH of neutralization	pH 5.0	pH 7.5
Temperature of fatliquoring	110°F.	135°F.
Time of fatliquoring	30 min.	90 min.
Time on horse before drying	1 day	10 days
Temperature in drying stove	ca. 100°F.	ca. 170°F.
Time in drying stove	12 hr.	24 hr.
Storage temperature	32°F.	77°F.
Helative humidity in storage	33% R.H.	77% R.H.

The skins used were processed to the "blue" stage in one yard. Only one fatliquor (sulfated sperm) was used. As a subsidiary investigation some of the same skins were sent to five different tanneries to be fatliquored with the same fatliquor and finished off as customary in the particular tannery. After the skins had been stored for fifty days and observations had been made of the spue upon them, various modifications of storage conditions were made and the spue assessments continued at intervals up to 156 days storage in all. Measurement were later made of the run of each skin. The results show that none of the factors varied in the processing is a dominant one leading to spue formation. If any modification of processing can, in itself, determine whether spue will appear in large or small quantities, the results of this investigation indicate that it will be found in the earlier stages, i.e. before the "blue" stage. The work suggests that spue formation is enhanced, but not to a very marked extent, if the period of drying in the stove is short, and if the drumming in fatliquor is prolonged. It is established that spue formation is aggravated by certain conditions of storage. Thus, high humidity increases spue, although a humidity as low as 33 per cent does not prevent it. If the storage temperature is constant, as much spue forms at 77°F. as at 32°F. Nevertheless, if skins are stored at 32°F. until they produce spue and are then transferred to a warmer atmosphere, the spue disappears temporarily. This occurs although the same skins will show spue if stored throughout the same warm atmosphere and although the spue, after vanishing at the higher temperature, reappears later at the same temperature. Somewhat greater run is obtained by the more thorough neutralization and by drumming in fat liquor for the longer period. Since the main experiment showed that the storage temperature and humidity were much more influential than other factors and these factors were uncontrolled in the cooperating tanneries, no information was obtained from the subsidiary experiment.

R. H. T.

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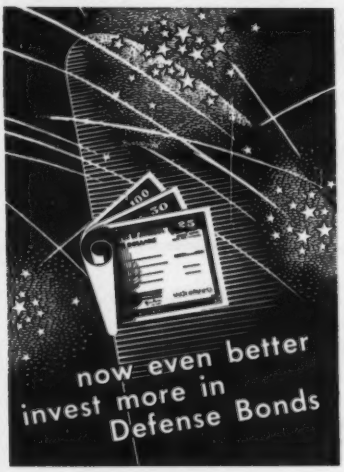

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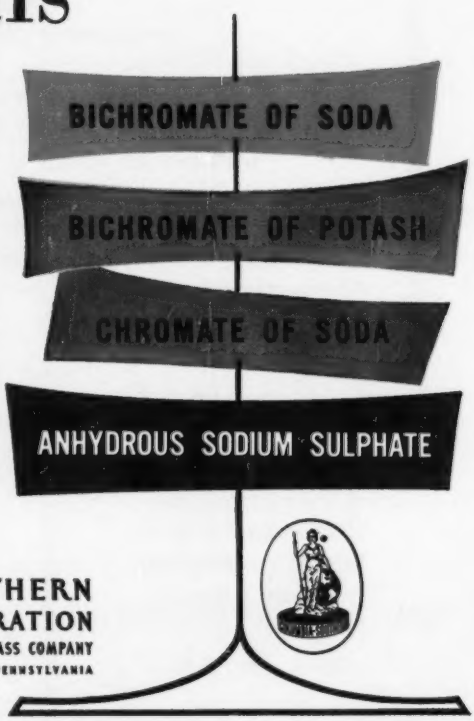

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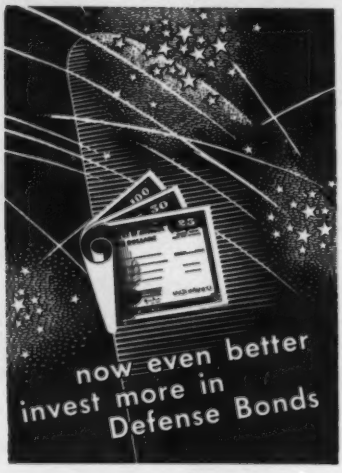

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ITALIAN
CHESTNUT EXTRACT

Solid — Powder — Spray Dried

also

MELLOWED CHESTNUT

Highly Soluble — Weight Giving — Good Color

MADE FROM LIVE WOOD

TYPICAL A.L.C.A. ANALYSIS

	Solid	Mellowed Powder	Spray Dried
TANNIN	61.72	65.95	73.5
NON-TANNIN	21.65	26.67	19.1
INSOLUBLES	.65	.67	.7
WATER	15.98	6.71	6.7

STAINLESS SUMAC CRYSTALS

HIGHEST QUALITY—MADE UNDER THE SUPERVISION OF THE WORLD'S LEADING EXTRACT CHEMISTS

SOLE AGENTS

TANEXCO, INC.

549 W. Washington Blvd. Chicago, Illinois

MYRTAN

A TANNING EXTRACT

made from the wood of an Australian species of the Eucalyptus family. It contains both catechol and pyrogallol tannin, the latter predominating.

TYPICAL A.L.C.A. ANALYSIS

	Ordinary	Clarified
TANNIN	61.55	64.34
NON-TANNIN	16.03	17.14
INSOLUBLES	1.70	0.72
WATER	20.72	17.80

Also Available as Liquid with 30% Tannin

As a retanning agent for chrome tanned leather, Myrtan possesses many advantages, such as smooth and strong grain, good color and break, as well as a round feel.

Blended into sole leather tannages, it has given better yields and improved color.

For further information address:

TANEXCO, INC.

549 WEST WASHINGTON BLVD., CHICAGO 6, ILLINOIS

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OAK
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Established 1887
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